OMC®

OUTBOARD MARINE CORPORATION

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July 8, 1999

Mr. Michael E. Bellott
United States Environmental Protection Agency
77 W. Jackson Blvd., SR6J
Chicago, IL 60604

Dear Mr. Bellott:

Please find enclosed copies of the Health and Safety Plan (HASP) and the revised Quality Assurance Project Plan (QAPP) for the Waukegan Harbor Remedial Action/Waukegan Harbor Superfund Site, Waukegan, Illinois.

The Operations and Maintenance Plan (O&M Plan) is currently being revised and will be provided separately.

If you have any questions or comments regarding the enclosed documents, please contact me at (847) 689-7046.

Sincerely,

Michael W. Rehor

Michael W. Rehor, P.E.
Project Manager

MR/lms
Enclosures

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN FOR OPERATIONS AND MAINTENANCE

QUALITY ASSURANCE PROJECT PLAN

ORIGINAL

FOR

OPERATION AND MAINTENANCE
WAUKEGAN HARBOR SUPERFUND SITE
WAUKEGAN, ILLINOIS

PREPARED FOR

WAUKEGAN HARBOR TRUST
WAUKEGAN, ILLINOIS

JANUARY 1999

BCM PROJECT NO. 00-8094-01

PREPARED BY

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ORIGINAL

QUALITY ASSURANCE PROJECT PLAN
SIGNATURE PAGE

FOR

OPERATION AND MAINTENANCE
WAUKEGAN HARBOR SUPERFUND SITE
WAUKEGAN, ILLINOIS

EPA Region V Quality Assurance Reviewer	Date
EPA Region V Remedial Project Manager	Date
Michael W. Rehm	3/31/99
OMC Project Coordinator	Date
Mark Gravler	1/27/99
BCM Engineers Inc. Quality Assurance Officer	Date
Arlyn S Allmendinger	6/29/99
Waukegan Harbor Trust Project Coordinator	Date
[signature]	3-11-99
Kemron Environmental Services Laboratory Manager	Date

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	-	Degrees Centigrade
COC	-	Chain-of-Custody
DQO	-	Data Quality Objective
EPA	-	Environmental Protection Agency
GC	-	Gas Chromatograph(y)
GC/MS	-	Gas Chromatography/Mass Spectroscopy
IEPA	-	Illinois Environmental Protection Agency
Kemron	-	Kemron Environmental Services
LIMS	-	Laboratory Information Management System
LQAP	-	Laboratory Quality Assurance Plan
LSC	-	Laboratory Sample Custodian
MDL	-	Method Detection Limit
MS	-	Matrix Spike
MSD	-	Matrix Spike Duplicate
ND	-	Non-detect(able) or Not Detected
NEIC	-	National Enforcement Investigations Center
NTU	-	Nephelometric Turbidity Unit
O&M	-	Operation and Maintenance
OMC	-	Outboard Marine Corporation
PARCC	-	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB(s)	-	Polychlorinated Biphenyl(s)
QA	-	Quality Assurance
QAMS	-	Quality Assurance Management Staff
QAPP	-	Quality Assurance Project Plan
QC	-	Quality Control
RAQAPP	-	Remedial Action Quality Assurance Project Plan
RL	-	Reporting Limit
RPD	-	Relative Percent Difference
RSD	-	Relative Standard Deviation
SAP	-	Sampling and Analysis Plan
SD	-	Standard Deviation
SOP(s)	-	Standard Operating Procedure(s)
Trust	-	Waukegan Harbor Trust
µg/L	-	Micrograms per Liter

LIST OF REFERENCES

1. "Remedial Action Quality Assurance Project Plan," written by Canonie Environmental Services Corp. for the Waukegan Harbor Superfund Trust, March 29, 1991.

2. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," EPA QAMS 005/80.

3. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA SW-846, Third Edition, November 1990, and Promulgated Updates.

4. Waukegan Harbor Consent Decree

5. "Specification and Guidance for Contaminant-Free Sample Containers," OSWER, Directive #9240.0-05A, December 1992.

6. "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA 600/1-80-018.

7. "NEIC Policies and Procedures," EPA-330/9-78-001-R, Revised August, 1991.

8. OMC Corporate Record Retention Policy

1.0 PROJECT DESCRIPTION

1.1 INTRODUCTION

This document is a Quality Assurance Project Plan (QAPP) for the Operation and Maintenance (O&M) of three containment cells at the Waukegan Harbor Superfund Site in Waukegan, Illinois. The O&M QAPP has been written by BCM Engineers Inc. for the Outboard Marine Corporation (OMC) and the Waukegan Harbor Trust (Trust). The O&M QAPP is intended to supplement the O&M Plan and to update site information previously provided to the Trust by Canonie Environmental Services Corp. on March 29, 1991 in the Remedial Action Quality Assurance Project Plan (RAQAPP). The focus of the O&M QAPP is to address sampling and analytical activities for monitoring the three water treatment systems (each dedicated to a particular Containment Cell) and for the monitoring of groundwater in and around the Slip No. 3, the West and the East Containment Cells, as identified in the O&M Plan.

The O&M QAPP is based on general information and conditions as described in the RAQAPP, as well as other previous site documents. The O&M QAPP was prepared following the Environmental Protection Agency (EPA) Quality Assurance Management Staff (QAMS) 005/80 guidance document, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans."

The purpose of the O&M QAPP is to indicate the prime responsibilities and prescribe the necessary procedures to determine that the project is executed in a manner consistent with generally accepted and approved quality assurance (QA) objectives and quality control (QC) procedures. The O&M QAPP identifies procedures for generating data that are precise, accurate, representative, comparable, and complete. This will allow project decisions based on analytical data to be made on an informed and logical basis.

The requirements of the O&M QAPP are applicable to the activities of all participants involved in the O&M work at the site. The O&M QAPP will address anticipated activities necessary to monitor the concentrations of polychlorinated biphenyls (PCBs) in the groundwater at specific locations in and around the Containment Cells and in the three Containment Cell water treatment systems designed to remove contaminants from the water.

All sections, figures, tables, and attachments referenced herein can be found in this document. Other documents that are referenced are identified in the References section of this O&M QAPP. If warranted, this O&M QAPP may be revised to include more information or alternate site-specific QA/QC procedures, as deemed necessary by the OMC Project Manager.

1.2 SITE BACKGROUND

Project background information is provided in the O&M Plan (this O&M QAPP is Appendix A of the O&M Plan). Sampling and analysis of the process influent, lead carbon and effluent streams from the three water treatment systems and of the groundwater from the monitoring wells surrounding the three Containment Cells (built after the completion of the remedial action construction activities), were not specifically addressed in the RAQAPP. This O&M QAPP will address those sample locations, sampling activities, and project data needs.

1.3 PROJECT OBJECTIVES/SCOPE OF WORK

The O&M Plan describes a regular operation, inspection, maintenance, and monitoring program to demonstrate the effectiveness of the remedial action. Specific operation and maintenance activities involve the collection and analysis of environmental samples, and therefore require defined quality assurance protocols. Quality assurance activities to be performed at the site in support of the operation and maintenance include the following:

- Periodic (as necessary) sampling of groundwater monitoring wells;
- Periodic (as necessary) sampling of water treatment system influent, lead carbon and effluent streams;
- Analysis of samples for PCBs following current EPA-approved methods;
- Monitoring of water levels in the monitoring wells, extraction wells and piezometers;
- Evaluation of the analytical results to ensure compliance with the Consent Decree.

1.3.1 Specific Sampling Tasks and Project Target Parameters

Analytical data will be collected to make informed and logical decisions that are consistent with the stated objectives for this project. For this project, it will be necessary to analyze samples from monitoring wells for PCBs to monitor the effectiveness of the Containment Cells. It will also be necessary to analyze samples from the water treatment systems to verify performance and to replace system components, when necessary.

The identified samples will be extracted for PCBs following SW-846 Method 3510B and analyzed for PCBs following SW-846 Update III Method 8082. The specific list of target parameters for this project is identified in Table 1, along with the corresponding reporting limits that will be provided by the laboratory for aqueous samples in order to meet the project objectives.

1.4 INTENDED DATA USAGE/DATA QUALITY OBJECTIVES

Site data have been generated and the results have been used to identify PCB concentrations in the groundwater at the site and to monitor the effectiveness of the water treatment systems. The data that will be generated in support of the O&M Plan will be compared with the regulatory objectives established for site cleanup and for public health protection. Additionally, the data will be used by the Trust to monitor the effectiveness of the Containment Cells and the water treatment systems, and to evaluate the potential impact to groundwater outside the three Containment Cells.

The data shall also be used by EPA Region V and/or the Illinois EPA (IEPA) to oversee site monitoring activities.

1.5 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale are based upon the requirements defined in the O&M Plan. Monitoring well locations are identified on Figure 1. Water treatment system sample locations are identified on Figure 2. Each task and sample matrix that requires analysis has defined sampling and analysis criteria that must be met to fulfill the requirements of the project. Sample matrices, sample locations, sampling frequencies, and analytical parameters are identified in Table 2, along with the frequencies of QA/QC samples that are associated with each sample matrix.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 OUTBOARD MARINE CORPORATION PROJECT PERSONNEL

The following is a list of key OMC project personnel and their responsibilities:

Project Principal-in-Charge:	Roger Crawford Responsible for project oversight and coordination.
Project Manager:	Michael Rehor Responsible for project management, subcontractor and laboratory coordination, and data analyses.
Sampling Coordinator(s):	Anthony Montemurro Responsible for sample collection, chain of custody documentation, and laboratory coordination.
Quality Assurance Manager:	Mark Traxler (BCM Engineers Inc.) Responsible for performing data review and for monitoring adherence to O&M QAPP specifications.

Project personnel may change, and such changes shall be reported to EPA Region V as needed, in conformance with the Consent Decree. Such staffing changes, however, do not require a modification, re-review, or additional approval of this O&M QAPP.

2.2 LABORATORY PERSONNEL

The analytical laboratory that will support the O&M activities will be the Kemron Environmental Services (Kemron) facility located in Marietta, Ohio. The following key laboratory personnel will have the responsibility for each item listed below the identified position.

2.2.1 Laboratory Project Manager

The Laboratory Project Manager will report directly to the OMC Project Manager and will be responsible for:

- Determining that resources of the laboratory are available on an as-required basis;
- Coordinating and scheduling laboratory analyses;
- Supervising in-house chain-of-custody;
- Overseeing data review and preparation of final reports;

2.2.2 Laboratory Quality Assurance Officer

The Laboratory QA Officer has the overall responsibility for data quality. The Laboratory QA Officer will be independent of the laboratory data generation, but will communicate data issues through the Laboratory Project Manager. In addition, the Laboratory QA Officer will:

- Overview laboratory quality assurance and QA/QC documentation;
- Conduct detailed data review;
- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures;
- Prepare Laboratory Standard Operating Procedures (SOPs).

2.2.3 Laboratory Sample Custodian

The Laboratory Sample Custodian (LSC) will report to the Laboratory Project Manager. Responsibilities of the LSC will include:

- Receiving and inspecting the incoming sample containers;
- Recording the condition of the incoming sample containers;
- Signing documents and verifying correctness of each chain-of-custody;
- Notifying the Laboratory Project Manager of sample receipt and inspection;
- Assigning a unique identification number and customer number, and entering each into the sample receiving log;
- Initiating transfer of the samples to appropriate lab sections;
- Controlling and monitoring access and storage of samples and extracts.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

3.1 DATA QUALITY PROTOCOLS

The overall quality assurance objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and data reporting that will provide results that are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance on field and laboratory equipment, and corrective action are described in other sections of the O&M QAPP.

To achieve data requirements that are consistent with the objectives of each task, there must be an assessment of the performance of five data quality parameters. These data quality parameters are precision, accuracy, representativeness, completeness, and comparability (PARCC). Definitions of these data quality parameters are presented below:

1. Precision -- A measure of the reproducibility of measurements under a given set of conditions;

2. Accuracy -- A measure of the closeness of a result compared to the true value.

3. Representativeness -- The degree to which a single measurement is indicative of the characteristics of a larger sample or the degree to which the data gathered by the project represent the field conditions.

4. Completeness -- A measure of the amount of valid data from the measurement system compared to the amount that is planned. Completeness is also defined as:

$$\% \text{ Completeness} = \frac{\text{Number of Valid Results}}{\text{Total Number of Results}} \times 100$$

Valid results will be defined for each task by addressing precision, accuracy, and representativeness parameters quantitatively.

5. Comparability -- A measure of the confidence with which one data set can be described as similar to another.

If the corrective actions provided for in this document are insufficient, the Project QA Manager will be contacted for resolution of the problem.

Data quality protocols will include the criteria specified by the referenced analytical methods. Data quality protocols may include, but not be limited to, analytical data quality requirements (QC acceptance criteria), data quality assessments and subsequent qualifiers, and required analytical documentation.

3.2 GENERAL OBJECTIVES

The quality of measurements made and data acquired will be determined by the PARCC characteristics. Specific objectives for each characteristic are established based on site conditions, objectives of the project, and knowledge of available measurement systems. The subsequent use of these measurements in calculations and evaluations is also subject to the guidelines set by the O&M QAPP as described in the following sections.

Subsequent to data review, any data points deemed to be unusable will be evaluated to determine their importance in meeting the project goals. The OMC Project Manager will discuss the unusable data points with the Project QA Manager to determine if the data should be rejected and resampling is required.

3.3 SPECIFIC DATA QUALITY OBJECTIVES

Specific data quality objectives (DQOs) have been established for this project to develop a data collection design that ensures data of sufficient quality and quantity to support defensible decision making. The project-specific DQOs are defined below.

3.3.1 Precision

Precision refers to the level of agreement among repeated measurements of the same parameter. It is usually stated in terms of standard deviation, relative percent difference (RPD) or relative standard deviation (RSD). The overall precision of a data point is a combination of sampling and analytical factors. Analytical precision is easier to quantify and control because the laboratory is designed to be a controlled, measurable environment. Sampling precision is unique to each site, making it more difficult to control and quantify. The goals for each factor are addressed here separately.

3.3.1.1 Field Precision

Field precision will be monitored by obtaining a duplicate sample at a minimum frequency of 1 per 10 samples collected for each sample matrix. Precision will be evaluated by calculating the RPD as follows:

$$RPD = \frac{\text{Difference Between the Two Measured Values}}{\text{Average of the Two Values}} \times 100$$

The RPD will be calculated for each analytical parameter. It is expected that positive aqueous duplicate results will have RPDs of less than, or equal to, 35 percent. If this criterion is not met, a careful examination of the sampling techniques, sample media, and analytical procedures will be conducted to identify the cause of the high RPD and to evaluate the usability of the data.

3.3.1.2 Laboratory Precision

Laboratory precision is laboratory- and analyte-specific and is evaluated by the analysis of duplicate samples. Laboratory precision is addressed in the Laboratory SOPs in Attachment 1. Acceptance criteria for precision for this project are identified in Table 3.

3.3.2 Accuracy

Accuracy refers to the difference between a measured value for a parameter and the true value for the parameter. It is an indicator of the bias in the measurement system. Sources of error measured by this parameter include the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analytical technique.

3.3.2.1 Field Accuracy

Field accuracy will be assessed by collecting an equipment rinsate blank or a field blank at a minimum frequency of 1 per 20 samples for each type of media that is collected. The accuracy goal for the equipment rinsate blanks and field blanks will be that they contain less than the method detection limit (MDL) or reporting limit (RL) concentrations for each parameter. If any analytes are detected in the blanks above these levels, the sample data will be compared with the blank data and may be rejected or qualified, based on the relative amounts present.

3.3.2.2 Laboratory Accuracy

Laboratory accuracy is laboratory- and analyte-specific and is evaluated by the analysis of laboratory blanks, surrogates, matrix spikes, and reference samples. Laboratory accuracy is addressed in the Laboratory SOPs in Attachment 1. Acceptance criteria for accuracy for this project are identified in Table 3.

3.3.3 Representativeness

Representativeness is the degree to which a set of data accurately and precisely relates to a characteristic of a population, parameter or condition. In sampling, the characteristic of representativeness is achieved by acquiring an aliquot of a larger mass, in a manner such that the aliquot possesses the same qualities, properties, and attributes as the mass from which it was taken. Sampling protocols will be utilized to assure that samples collected are representative of the media present in the field. Sample handling protocols including such tasks as storage, transportation, and preservation have been developed to protect the representativeness of the samples gathered during the project. Proper documentation in the field and in the laboratory will

establish that protocols designated to preserve the representative mass of the samples have been followed, and sample identification as well as sample integrity has been preserved. If deviations from the O&M QAPP occur, the OMC Project Manager and the Project QA Manager will be notified for direction. Changes will be documented and provisions will be made to ensure that the changes do not reduce the representativeness of the samples to the overall field conditions.

3.3.4 Completeness

Completeness is a measure of the amount of valid data obtained, compared to the amount that was specified to be obtained under normal conditions. The percentage of valid data that is considered acceptable is established based on the measurements required to accomplish the specific project DQOs. The extent of completeness must be reviewed on a relative basis for sample collection activities, since the required amount of valid data anticipated prior to sampling episodes may not accurately define the amount of data ultimately necessary to render a correct decision. A certain amount and type of data must be collected during the site monitoring activities for conclusions to be valid. Missing data may reduce the precision or accuracy of estimates or introduce bias, thus lowering the confidence level of the conclusions.

The importance of any lost or suspect data will be evaluated in terms of the sample location, analytical parameter, nature of the problem, decision to be made, and the consequence of an erroneous decision. Critical locations or parameters for which data are determined to be inadequate or rejected may be resampled and reanalyzed.

The goal for completeness is 100 percent. After the collection, analysis, and evaluation of all sample data are done, and all critical sample results are addressed and complete, a value of 95 percent completeness will be considered acceptable as a DQO for the remaining non-critical data.

3.3.5 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability reflects both the internal consistency of data collected with regard to a single parameter and an expression of data in units that are consistent with the units which data gathered by other organizations measuring the same parameter are presented. Comparability of data gathering and measuring procedures should also be addressed if data are to be reliably compared. Thus, the characteristic of comparability implies the personnel involved in data acquisition and reduction must operate measurement systems within the calibrated range of the particular instrument as well as utilize analytical methodologies that produce comparable results.

When a comparison of data sets identifies certain values within one or more sets that are not consistent with the totality of the data acquired, these values, known as "outliers," must be reassessed prior to utilization in the decision-making process. Utilization of statistical analysis is often required to define whether the outliers represent significant values that require rejection or qualification in the decision-making process. If duplicate analysis of samples verifies that an outlying value is valid, additional sampling and analysis may be conducted to determine the true characterization of the media being sampled.

3.4 LEVEL OF QUALITY CONTROL EFFORT

Field blank, equipment rinsate blank, method blank, field duplicate, and matrix spike/matrix spike duplicate (MS/MSD) samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field blank or equipment rinsate blanks may be submitted to the analytical laboratories to provide the means to assess the quality of data resulting from the field sampling procedures. Field blank samples are analyzed when only dedicated sampling equipment are used to check for procedural contamination due to sample collection and shipment. Equipment rinsate blank samples are analyzed when any non-dedicated sampling equipment are used to check for procedural contamination, including the potential for cross-contamination. Method blank samples are generated within the laboratory and are used to assess potential contamination resulting from laboratory procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes/matrix spike duplicate samples provide information about the effect of the sample matrix on the sample preparation (extraction) and measurement methodology.

There are two general levels of QC effort for field sampling activities, depending on the type of sample collected and the intended use of the data generated.

For groundwater samples, the QC effort for each sampling event will include at least 1 equipment rinsate blank for every 20 or fewer groundwater samples when any non-dedicated sample collection equipment is utilized (or 1 field blank for every 20 or fewer samples when **only** dedicated sample collection equipment is utilized); 1 field duplicate for every 10 or fewer groundwater samples; and 1 MS and 1 MSD for every 20 or fewer groundwater samples (these QC samples are not required for waste water sampling events).

For waste water samples, the QC effort is less stringent and is based on a running total for all waste water sampling events combined. The overall QC effort for waste water samples will include at least 1 equipment rinsate blank for every 20 waste water samples when any non-dedicated sample collection equipment is utilized (or 1 field blank for every 20 samples when **only** dedicated sample collection equipment is utilized); and 1 field duplicate for every 10 waste water samples.

The level of QC effort provided by the laboratory will, at a minimum, meet the requirements of the referenced analytical methods. The estimated quantities of field samples and QC samples for each sample matrix are identified in Table 4.

4.0 SAMPLING PROCEDURES

4.1 PRESAMPLING PREPARATION

Before sampling, presampling preparation protocols will be followed as described in the following sections. These protocols will include the use of cleaning materials and analyte-free water, sampling equipment decontamination, the use of appropriate sample containers, appropriate sample preservation, and field sample documentation.

4.1.1 Cleaning Materials and Analyte-Free Water

Laboratory-grade detergent will be a standard brand of non-phosphate detergent such as Alconox® or Liquinox®. The standard decontamination solvent will be pesticide-grade hexane. Demonstrated analyte-free water will be used as the final water rinse.

The laboratory must maintain QC records demonstrating, on a regular basis, that no detectable concentrations of target compounds exist in the analyte-free water that is used by the samplers for decontaminating equipment and for collecting QC blank samples. If the analyte-free water is obtained by a source other than the laboratory, OMC will maintain QC records identifying the source and verifying that no detectable concentrations of target compounds has been identified in the water that is used.

4.1.2 Sampling Equipment Decontamination Procedures

In order to prevent possible contamination from sampling equipment, all non-dedicated sampling devices will be decontaminated prior to sample collection activities. Whenever possible, sampling equipment will be dedicated to one sampling location. When it is not possible to dedicate sampling equipment, field decontamination will be performed between each sampling location.

All sampling equipment will be decontaminated according to the following procedure:

1. Wash and scrub with a non-phosphate detergent plus tap water wash;
2. Tap water (or demonstrated analyte-free water) rinse thoroughly;
3. Demonstrated analyte-free water rinse;
4. Solvent (hexane) rinse twice;
5. Total air dry.

Sampling equipment should be washed and scrubbed with a phosphate-free detergent and rinsed with tap water or demonstrated analyte-free water in the field as soon as possible after use.

4.1.3 Sample Containers

Sample containers will be obtained from the laboratory throughout the project. The type and size of containers, the required preservatives, and the maximum holding times for each analysis are identified in Table 5. The laboratory will provide pre-cleaned containers in accordance with the EPA document "Specification And Guidance For Contaminant-Free Sample Containers," OSWER, Directive #9240.0-05A, December 1992 and will maintain records to verify that the containers were shown to be free from contamination.

4.1.4 Sample Preservation

All samples will be preserved in accordance with the requirements of "Test Methods for Evaluating Solid Waste," SW-846, Third Edition, with promulgated updates. Specific sample preservation requirements are identified in Table 5. Samples will be preserved in the field immediately after collection and submitted to the laboratory as soon as possible (no later than 72 hours after sample collection).

4.2 SPECIFIC SAMPLING PROCEDURES

4.2.1 Groundwater Sampling Procedures

Groundwater sampling will be conducted following the Field Sampling SOP entitled "Groundwater Sampling" in Attachment 2. The samples that will be collected and the analyses that will be conducted are identified in Table 2.

4.2.2 Waste Water Sampling Procedures

Waste water (water treatment system) sampling will be conducted following the Field Sampling SOP entitled "Waste Water Sampling" in Attachment 2. The samples that will be collected and the analyses that will be conducted are identified in Table 2.

4.2.3 Other Sampling Procedures

If sampling of a specific medium is required and not addressed by procedures specified in this O&M QAPP, sampling procedures outlined in "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA 600/1-80-018, will be followed in the collection of samples from various media at the site. If modification of the procedures provided herein or required by EPA 600/1-80-018 are necessary, the OMC Project Manager will prepare written sampling procedures prior to collection of the sample and submit the procedures to EPA Region V for approval. Split or duplicate samples, in appropriate containers accompanied by a sample label and a chain-of-custody form, will be provided to EPA Region V upon request.

4.3 FIELD DOCUMENTATION/FIELD LOG BOOKS

Health and safety activities and field sampling activities will be recorded in a field log book or on designated forms. Field log books will be permanently bound, and each page will be numbered, dated, and signed by the person making the entry. Designated forms will also be dated and signed by the person making the entry. All entries should be made in black ink. Errors should be crossed out with a single line, initialed, and dated. At the completion of the day, if a page in a field log book is not complete, a diagonal line will be drawn through the remainder of the page with the signature at the bottom, indicating the conclusion of the entry or the day's activities.

A system of logging all pertinent data collected during sampling activities will be maintained using bound field log books or designated sample log forms. All sample locations will be recorded and may be referenced to the site map so that each location is permanently established. Samples will be tagged or labeled with all pertinent site information at the time of sampling.

4.4 SAMPLE NAMING CONVENTION

Unique sample names for each sampling location utilize the following format:

A(A)B(B)-C

Where:

A(A) = General sample type or location (i.e., W = Monitoring Well, S3T5 = Slip 3 Containment Cell [Category 5 Treatment System], E3T5 = East Containment Cell, W3T5 = West Containment Cell)

B(B) = Specific well number or process location (i.e., 1, 2, 3, 4, 5, 6,...to 12, or I = Influent, LC = Lead Carbon, E = Effluent)

C = QC identifier, as needed (i.e., D or DUP = Duplicate, E = Equipment Rinse Blank, F = Field Blank, M or MS = MS, N or MSD = MSD, T = Trip Blank)

Sampling locations shall be referred to by the sample identification number on all submittals.

5.0 SAMPLE CUSTODY

A laboratory sample is utilized as physical evidence regarding a site. Due to the evidentiary nature of the data collected, the possession of these samples must be traceable from the time the empty sample containers are prepared by the container supplier through the reporting of the analysis results

As an essential part of project management, OMC has established sample control procedures to ensure sample integrity. All sample containers and samples will be maintained under strict custody procedures throughout the investigation. The sample control procedures are based on EPA National Enforcement Investigations Center (NEIC) protocols for bulk sample shipment.

An established program of sample chain-of-custody procedures that is followed during sample collection and handling activities in both the field and laboratory operations is required to maintain sample integrity. The program is designed to account for each sample at all times. Proper completion of field sample logs, sample tracking sheets, sample extraction logs, and laboratory reports by appropriate field and laboratory personnel provide for thorough monitoring of the samples from the time of collection through analysis and final report generation.

The objectives of sample identification, custody, and monitoring procedures are to determine that

- Samples are uniquely labeled for identification purposes throughout the analytical process;
- Important sample characteristics are preserved;
- Samples are protected from loss, damage, or tampering;
- Any alteration of samples (e.g., filtration, preservation, or damage due to shipment or other processes) is documented;
- Samples are correctly analyzed;
- Results are traceable to field records; and,
- A record of sample integrity and analytical fate is established for legal purposes.

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files are maintained under document control in a secure area following procedures described in detail in Section 5.5.

In accordance with NEIC requirements, samples collected for chemical analysis and evidence files will be considered to be in a person's custody if any of the following conditions apply:

1. They are in actual possession of an authorized person;

2. They are in view of an authorized person, after being in his/her possession;

3. They were in the physical possession of an authorized person and then were locked up or sealed in a tamper-proof manner; or,

4. They are placed in a designated and identified secure area.

The Sampling Coordinator will review field activities daily to determine whether proper custody procedures were followed during field work and will determine if additional samples are required.

5.1 SAMPLE MONITORING FORMS

The use of specific forms accomplishes one or more of the specific objectives of sample custody, identification, or control. The use of each of the forms is discussed below.

The purpose of sample monitoring forms is to complement and document the verbal communication between the Sampling Coordinator and the laboratory chemists and technicians. Lines of communication between the field and the laboratory must remain open to inform the laboratory of upcoming sampling events, what will be sampled, the general character of the samples, and when results must be available. The field staff members must communicate with the laboratory to determine that samples are received, sufficient sample volume was provided, and sample documentation from the field is correctly interpreted by the laboratory staff.

During sampling, Sampling Coordinator should note any observed characteristics which may provide information about the type or concentration of target compounds to expect in the sample.

5.1.1 Field Sample Log Form

The field sample log form is completed in the field by the Sampling Coordinator. The field sample log form correlates the assigned sample identification number to a specific sample location. An example of a field sample log form is presented on Figure 3.

5.1.2 Sample Label

The sample label on each sample container will be completed in its entirety to ensure that pertinent information accompanies the sample to the laboratory. The sample label must be completed in the field by the Sampling Coordinator. Information on the sample label will include, at a minimum, the following:

- A unique sample identification number
- Sample description (sample type, matrix, or location)
- Date sampled
- Time sampled
- Sampling Coordinator's name(s)
- Analyses requested
- Any relevant comments (such as readily detectable or identifiable odor, color or known toxic properties)

Complete information should be printed in waterproof ink or indelible marker on each label, so that if any sample container is separated from the rest of the shipment in the laboratory, sufficient information is available on the label to ascertain the scheduled work required on the sample. The chain-of-custody form should reflect the identification of each sample for which laboratory results are requested. If samples must be held at the laboratory pending further instructions, an appropriate comment will be made on the chain-of-custody form. An example of an appropriate sample label is presented on Figure 4.

5.1.3 Chain-of-Custody Record

The chain-of-custody (COC) record form is completed in the field by the Sampling Coordinator(s). The COC form may be completed in conjunction with the field sample log form or prior to the shipment of samples to the laboratory. The COC form contains information on the date and time of sample collection, the name of the Sampling Coordinator(s), the project name, the number and type of containers for each sample shipped, and an itemization of the analyses requested for each sample, together with any remarks about the sample prior to shipment. The COC form is enclosed with the samples after it has been signed by the Sampling Coordinator(s). The COC form is then signed each time possession of the samples changes, with the signature of the persons relinquishing and receiving the sample. The date and time of exchange must also be indicated on the record. An example of a COC form is presented on Figure 5.

The COC form will serve as the formal custody record and request for analysis. This form may be revised to meet field conditions, as necessary. Chain-of-custody forms will be executed and retained as follows:

- One copy retained by the Sampling Coordinator or OMC Project Manager
- Original sent with samples to the analytical laboratory

The LSC will inventory each shipment of samples to confirm that the integrity of the samples and containers has not been compromised and then sign and date the original COC form. The LSC will then make a note on the custody form of any discrepancy in the number of samples or breakage of samples. The LSC must notify the Sampling Coordinator immediately of any problems identified with shipped samples. The LSC must provide a copy of the completed COC form for the analytical results package for each sample.

5.2 FIELD CUSTODY PROCEDURES

In the field, samples should be handled by as few people as necessary to ensure sample integrity. The following procedure will be used to track custody of containers and samples from the point of distribution through laboratory receipt of samples:

1. Empty, pre-cleaned containers will be supplied by the laboratory to the Sampling Coordinator at the site.

2. Each sample collected will be entered on the chain-of-custody record form.

3. The original COC form will accompany the sample containers during transport to document their custody.

4. When custody is relinquished to a common parcel carrier for delivery to the laboratory, the following protocol will be followed:

    a. The original COC form will be placed inside the shipping package.

    b. The shipping package will be sealed with strapping tape. The tape will be placed on the package in such a manner that the package cannot be opened without breaking the tape. The tape shall be signed and dated by the Sampling Coordinator. The tape will serve to document that the samples remained unaltered during shipment through the common parcel carrier.

6. The laboratory will assume custody of the samples upon receipt and the LSC will be charged with sample care and receipt following laboratory custody procedures as described in detail in Section 5.4. All samples will be logged in a bound, volume-numbered accession logbook or a Laboratory Information Management System (LIMS) to track sample information.

7. The laboratory will retain custody of the samples in a secure area until such time as the samples are destroyed.

8. The following information must be supplied to complete the COC form:

   a. Project name (Waukegan Harbor);

   b. Signature of Sampling Coordinator;

   c. Sample field identification, date and time of collection, and grab or composite sample designation;

   d. Signatures of individuals involved in sample transfer. Individuals receiving the samples shall sign, date, and note the time that they received the sample on the COC form;

   e. In the lower right comment section, the type of carrier service will be indicated; and,

   f. The total number of sample containers must be listed in the indicated space for each sample.

5.3 SAMPLE PACKING AND SHIPMENT PROCEDURES

At the end of each sampling event, all samples will be packed in shipping containers for overnight delivery to the analytical laboratory. Sample packing and shipping procedures are listed below.

1. Each container will be checked for a properly completed sample label.

2. Each sample bottle will be placed in padded divider or in a zip-lock plastic bag. Allow sufficient space in all bottles to compensate for any pressure or temperature changes (approximately ten percent by volume). Ensure that the lids on all bottles are tightly sealed.

3. A large capacity cooler or specific laboratory-prepared sample shipping container will be used to ship the samples. Each cooler will have the drain plug taped shut (inside and out). Approximately one to two inches of packing material should be placed in the bottom of the liner, if necessary.

4. Sample bottles will be placed in the shipping container and secured with packing material, as necessary, to ensure that the containers will not shift in transport or directly contact each other.

5. Samples will be maintained at 2 to 6 degrees centigrade ($4 \pm 2°C$) during shipment. Ice that has been placed in heavy duty polyethylene bags and sealed, or cold packs (such as Blue Ice), will be used to cool the samples.

6. After all sample containers are packed, additional packing material will be added as needed to fill void space.

7. The associated COC form will be placed in a polyethylene bag, sealed, and placed inside of the shipping container.

8. The cooler or shipping container will be taped closed with strapping tape.

9. The tape will be applied in such a manner that the cooler cannot be opened without disturbing the tape.

10. The cooler will be relinquished to the courier with the required signed and dated hand bill.

11. Samples will be shipped from the field to the laboratory. All shipments will be accompanied by the COC form identifying the contents. The back copy will be detached in the field and kept as part of the field record.

5.4 LABORATORY CUSTODY PROCEDURES

Upon receipt in the laboratory, the cooler and all samples will be inspected thoroughly by the LSC to confirm that the integrity of the samples has not been compromised. The individual sample containers will be inspected to verify that each sample has a sample label. The condition of the samples will be noted on the COC form.

The sample containers will be checked against the accompanying COC form to verify that the cooler contents are identical to the samples described on the COC documents. If discrepancies exist, they will be reported to the Laboratory Project Manager, who will immediately notify the OMC Project Manager. The problem will be resolved, in writing, before analytical work begins.

The samples will be placed in a secured storage area, under the conditions called for by the analytical method, until taken for analysis.

5.5 PROJECT FILES

During the O&M activities, project files will be accumulated and assembled. Project files shall be maintained in secure filing cabinets in OMC's office during the implementation of this project and will be available for EPA Region V review. The project files will contain all of the information discussed below.

The project laboratory files, containing sample collection data consisting of the laboratory analytical data (sample logs, custody records, chromatograms, calibrations, QC results, computer printouts, sample preparation logs, etc.), will be maintained by the file custodian in filing cabinets or other secure manner. Copies of data files, log books, and other documentation generated by each laboratory may be provided to OMC, if requested, upon completion of all analytical work for the project. The original documentation shall be maintained by each laboratory in a secure location.

The project files may consist of, but not be limited to, the following:

- Laboratory data deliverables
- Sample collection data
- Field log books
- Field sample log forms
- Survey maps
- Site photographs
- Project correspondence
- Interim and final reports
- Drawings, specifications, and addenda
- Modifications to the contract
- Accepted special procedure plans
- Field test records
- Chain-of-custody documents
- Other items, as may be required by the OMC Project Manager

The project files will be maintained in the office in accordance with OMC's internal procedures for document retention.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.1 FIELD CALIBRATION

Field equipment calibrations will be performed before use. Calibrations will be performed as specified by the Field Analytical SOPs included in Attachment 3, and recorded in bound field log books or designated log forms. The following summarizes the calibration requirements:

1. Temperature will be measured using a thermometer that has been checked for accuracy. This will be accomplished by measuring the temperature of a mixture of distilled water and distilled ice that has been allowed to come to thermal equilibrium. The thermometer must read between -0.5 and 0.5 °C, (or between 31 and 33 °Fahrenheit) to be determined sufficiently accurate.

2. The pH meter will be calibrated daily in the field using known buffer solutions of 4.0 and 7.0, or 7.0 and 10.0 (depending upon the expected sample pH), with ambient temperature compensation. Documentation of calibrations will be made in instrument and/or field log books or designated sample log forms.

3. The conductivity meters are factory-calibrated. Meters will also be calibration checked daily in the field by the sampling personnel using the instrument red-line calibration and cell test functions.

4. Turbidity will be measured using a field-calibrated turbidimeter. The meter shall be calibrated using a 1.0 Nephelometric Turbidity Unit (NTU) standard and a 40.0 NTU standard, or other appropriate standards which bracket the sample readings. Documentation of calibrations will be made in instrument and/or field log books or designated sample log forms.

All results of field measurements will be recorded in bound field log books or designated sample log forms and will be incorporated into the project documentation files for reporting purposes. Daily calibration information should be maintained in bound field log books. The recorded information will include the date of calibration, the standards used, and the calibration results. Initial calibration and field calibration documentation will be available upon request.

6.2 LABORATORY CALIBRATION

Laboratory instruments will be calibrated following the referenced EPA-approved analytical method protocols in accordance with the Laboratory SOPs identified in Attachment 1. Initial calibrations will be performed before sample analysis, and continuing calibration checks will be performed at the frequencies specified in the corresponding Laboratory SOP.

7.0 ANALYTICAL PROCEDURES

7.1 FIELD ANALYTICAL PROCEDURES

Temperature, pH, specific conductance, and turbidity of the groundwater and water treatment system (waste water) samples will be analyzed onsite. The analyses of these parameters are necessary to ensure that proper well development has been accomplished and to document site conditions. Field Analytical SOPs are provided in Attachment 3.

7.2 LABORATORY ANALYTICAL PROCEDURES

PCBs will be extracted from aqueous samples following SW-846 Method 3510B and analyzed in accordance with SW-846 Method 8082.

The EPA-approved procedures and supporting report documentation have been selected for the sample analyses to supply the level of QC and documentation needed to characterize the groundwater and waste water samples adequately.

The analytical methods used for the project are identified in Table 1. The choice of analytical methods and the level of data quality outlined in Table 2 are considered sufficient to meet the project data quality objectives (DQOs) for the O&M for groundwater data and for waste water data.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

8.1 DATA REDUCTION

All results will be reported in the units specified by the analytical method referenced. Equations to calculate concentrations are also found in the method referenced. All blank results will be reported and will be used in data validation to review sample results qualitatively. Kemron data reduction and handling procedures are outlined in the Kemron Laboratory QA Manual.

All project documents, correspondence, data packages, and other project information will be stored and maintained in OMC's project file system located in Waukegan, Illinois for a minimum of six (6) years after the EPA has acknowledged completion of the work or the matter is otherwise fully resolved, in accordance with OMC's corporate policy for record retention.

8.2 DATA VALIDATION

8.2.1 Field Data Validation

Validation of the field data is the prime responsibility of the Sampling Coordinator. The following issues will be addressed:

- Proper COC, sample handling and delivery, and decontamination procedures are followed;
- Samples are collected following the specified methods;
- Field instrument calibrations are performed and properly recorded according to the specified methods;
- Field analyses are performed and recorded according to the appropriate Field Analytical SOPs;
- Quality control samples are collected, as required;
- Field log books are fully completed and in agreement with sample container labels and COC forms.

8.2.2 Laboratory Data Validation

Validation of the laboratory data is the prime responsibility of the laboratory supervisors and the Laboratory QA Officer. The following areas will be addressed:

- Chain-of-custody and sample handling procedures are followed;
- Holding times are met;
- Samples are prepared and analyzed according to the specified methods;
- Instruments are calibrated and documented as required;
- Quantitation/detection limits are determined and reported correctly;
- QC requirements are performed and within required ranges;
- Method blanks are prepared and analyzed as required;
- Calculations are performed correctly and verified;
- Completeness and transcription of raw and final data are correct.

8.2.3 Project Data Validation

The full deliverable data package requirements for groundwater samples specified in Section 8.3 will allow data review to be completed by the Project QA Manager using a Data Validation Checklist for each data package. Table 6 shows an example of a Data Validation Checklist. Project data validation is not required for water treatment system (waste water) data packages.

Items that will be verified for groundwater samples include COC documentation, holding times, initial and continuing calibrations, surrogate and spike recoveries, blank results, and historical data comparability to previous site results for each location.

8.3 DATA REPORTING

All full deliverable data packages from the laboratory for groundwater samples must be paginated in ascending order. The laboratory will keep a copy of the paginated package in order to be able to respond efficiently to data validation inquiries. Any reporting errors identified during the data validation process must be corrected by the reporting laboratory, as requested. All data validation inquiries to the laboratory must responded to by the laboratory quality assurance department.

At a minimum, the following data deliverables are required in each laboratory data package:

- Cover page, including laboratory name and address, laboratory certification number, date of analytical report, and signature of laboratory director;
- A listing of all field samples and corresponding laboratory sample numbers;
- A listing of the analytical methods used and detection limits for each analyte;
- Tabulated sample results, including date of analysis;
- Raw instrument printouts for samples, QC samples, and calibrations;
- Method blank and other QC results tabulation;
- Chain-of-custody documents.

9.0 INTERNAL QUALITY CONTROL CHECKS

9.1 ANALYTICAL QUALITY CONTROL SAMPLES

The analytical methods and Laboratory SOPs referenced address the QC procedures used and the required frequencies for analytical QC samples, including replicates, spike samples, blanks, calibration standards, internal standards, and surrogate spikes.

9.2 FIELD QUALITY CONTROL SAMPLES

9.2.1 Equipment Rinsate Blanks and Field Blanks

Two types of blanks may be collected in the field. When any non-dedicated sampling equipment is used to collect samples, equipment rinsate blanks will be collected as field QC samples. If only dedicated sampling equipment is used for sampling, field blanks will be collected instead.

Equipment rinsate blanks are recommended for sampling events when any non-dedicated field sampling equipment are used. Equipment rinsate blanks are collected by pouring demonstrated analyte-free water over the decontaminated sampling equipment as a check to ensure that the decontamination procedure has been adequately carried out and that no cross-contamination of samples is occurring due to the equipment itself. Analyses of equipment rinsate blanks are performed for all analytes of interest, as specified on Table 4. For groundwater samples, one equipment rinsate blank will be collected at a minimum frequency of 1 per 20 samples or one per sampling event, whichever is more frequent, for each type of non-dedicated sampling equipment used. For waste water samples, one equipment rinsate blank will be collected at a minimum frequency of 1 per 20 samples for each type of non-dedicated sampling equipment used.

Field blanks are recommended for sampling events when only dedicated field sampling equipment will be used. Field blanks consist of pouring demonstrated analyte-free water into sample containers for shipment to the laboratory as a check to ensure that there is no introduction of contamination in the sample collection or shipment process. Analyses of field blanks are performed for all analytes of interest, as specified on Table 4. For groundwater samples, one field blank will be collected at a minimum frequency of 1 per 20 samples or one per sampling event, whichever is more frequent, for each type of dedicated sampling equipment used. For waste water samples, one field blank will be collected at a minimum frequency of 1 per 20 samples for each type of dedicated sampling equipment used.

9.2.2 Field Duplicates

A field duplicate consists of an actual sample for which twice as much volume as necessary to fill all sample containers has been collected. Aliquots are then equally distributed in two sets of sample containers. This division results in two equal samples collected from one sampling location. Field duplicates are used to assess consistency of sampling, sample homogeneity, and laboratory analytical consistency. These sample duplicates may be submitted as laboratory blind duplicates and will be analyzed for all analytes of interest. Analyses of field duplicates are performed for all analytes of interest, as specified on Table 4. For groundwater samples, field duplicates will be collected at a minimum frequency of 1 per 10 samples, or one per sampling event, whichever is more frequent. For waste water samples, field duplicates will be collected at a minimum frequency of 1 per 10 samples.

9.2.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates are laboratory-spiked samples that are subjected to the same analytical procedures as the original sample collected in the field. Three aliquots of a sample are measured by the laboratory, two of these aliquots are spiked with known quantities of specific analytes, and all three are extracted and analyzed in order to measure and evaluate laboratory precision and accuracy. Analyses of matrix spike/matrix spike duplicates are performed for all analytes of interest, as specified on Table 4. For groundwater samples, matrix spike/matrix spike duplicates will be collected at a minimum frequency of 1 per 20 samples, or one per sampling event, whichever is more frequent. For waste water samples, site-specific matrix spike/matrix spike duplicates are not necessary.

For aqueous matrix samples, triple the normal volume for organic parameters and double the normal volume for inorganic analyses will be collected for matrix spikes. No additional volume will be required for non-aqueous matrix samples.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with procedures established in the O&M QAPP. The audits of field and laboratory activities may include two independent parts: internal audits conducted by OMC, and external audits conducted by the EPA.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 Internal Field Audits

An internal audit of field activities, including sampling and field measurements, will be conducted by the Project QA Manager or his designated representative at or near the beginning of the implementation of the approved O&M QAPP. The intent of the QA Audit is to verify that all established procedures that are documented in the O&M QAPP are being followed correctly.

The QA Audit will include an examination of field sampling records, field instrument operating records, sample collection frequencies and techniques, maintenance of QA procedures, and chain-of-custody documentation. A follow-up QA Audit may be required to document the correction of any deficiencies. During the field QA Audit, the Project QA Manager will review data handling procedures with the OMC Project Manager and Sampling Coordinators. Items such as accuracy, consistency, documentation, and adherence to the SOPs will be discussed.

10.1.2 External Field Audits

External field audits may be conducted by EPA Region V at any time. These audits may or may not be announced and are at the discretion of the EPA. External field audits will be accomplished through the observation of the sampling operations by the EPA Region V representative.

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Laboratory Audits

An internal laboratory audit may be conducted by the Project QA Manager, if necessary. An internal laboratory system audit would include an examination of laboratory documentation on sample receiving, log-in, storage and chain-of-custody procedures, sample preparation and analysis, and operational records. Performance audits may involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis. The Project QA Manager would evaluate the analytical results of theses blind performance samples to determine if the laboratory maintains acceptable performance.

10.2.2 External Laboratory Audits

An external audit may be conducted by the EPA QA Laboratory at any time. An external audit may be unannounced and is at the discretion of EPA Region V. The laboratory audit may include, but not be limited to, review of laboratory procedures, laboratory audits, and/or submission of standard reference material or performance evaluation samples to the laboratory for analysis.

10.3 AUDIT REPORTS

A written report of each QA Audit will be prepared to include the following:

1. An assessment of project status in each of the major project areas regarding QA/QC issues;

2. Clear statements of areas requiring improvement or problems to be corrected. Recommendation and assistance will be provided regarding proposed corrective actions or system improvements. If no action is required, the report will state that the QA audit was satisfactorily completed;

3. A time table for any corrective action required; and,

4. A follow-up to determine that recommendations have been implemented.

A QA Audit report will be written by the Project QA Manager within 15 days of the completion of auditing activities. The QA Audit report will be distributed to the OMC Project Manager, the Sampling Coordinator, and the Laboratory Project Manager. The QA Audit report will also be provided to EPA Region V in the regular quarterly report which documents the samples that were collected during the QA Audit.

11.0 PREVENTIVE MAINTENANCE

11.1 FIELD PREVENTIVE MAINTENANCE

All field instruments will be checked prior to use in the field. Field Analytical SOPs outlined in Attachment 3 specify the types and frequency of maintenance checks. All maintenance performed on field instruments will be recorded in a log book that is maintained by OMC.

11.2 LABORATORY PREVENTIVE MAINTENANCE

Preventive maintenance and periodic maintenance will be performed as needed and documented in the lab notebooks, instrument maintenance logs, or work orders, as appropriate, in accordance with EPA requirements. Preventive maintenance procedures and schedules for laboratory instruments are outlined in the Laboratory QA Manual. This manual may be supplied upon request.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS PRECISION, ACCURACY, AND COMPLETENESS

The purpose of data quality assessment is to determine that data generated under the program are accurate and are consistent with project objectives. The quality of groundwater data will be assessed based on the precision, accuracy, and completeness of the data that are generated for groundwater samples. The quality of waste water data will not necessarily be assessed.

Groundwater sample results will be assessed to determine if the selected procedures are efficient and effective, and that the data generated provide sufficient information to achieve the project objectives. Precision and accuracy of measurement systems will also be evaluated. In general, evaluation of data will be based on performance audits and review of completeness objectives.

Documentation for groundwater sample data will include the following, at a minimum:

1. Number of replicate samples collected;

2. Number of replicate and spike samples analyzed;

3. Use of historical data and its reference;

4. Identification of analytical methods used;

5. Evaluation of the data package, which may include the following:

    - Initial calibration and calibration verification;
    - Continuing calibration verification;
    - Spiked sample analysis;
    - Surrogate recoveries;
    - Blank summaries.

The laboratory is responsible for any additional evaluation required of analytical data packages.

Throughout data collection activities, an assessment of the adequacy of the database generated in regard to meeting the project objectives will be undertaken. Recommendations for improved QC will be developed, if appropriate. In the event that data gaps are identified, the Project QA Manager may recommend the collection of additional data to fully support the project's findings.

Specific QA objectives for precision and accuracy of surrogate recoveries and spike recoveries that will be followed by the laboratory for groundwater and waste water samples are located in the Laboratory SOPs in Attachment 1.

12.1 PRECISION ASSESSMENT

Spiked samples are prepared by selecting a sample at minimum specified frequencies, dividing the sample into three equal aliquots, and then spiking two of the aliquots with a known amount of analyte. These three samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The RPD between the spike and spike duplicate are calculated and plotted. The RPD is calculated according to the following formula:

Precision (for matrix spike samples):

$$\frac{S_1 - S_2}{[S_1 + S_2]/2} \times 100 = RPD$$

Where: S_1 = Larger Spike Value
S_2 = Smaller Spike Value
RPD = Relative Percent Difference

Duplicate samples are collected at minimum specified frequencies in the field and shipped to the laboratory as independent samples. These samples are then included in the analytical sample set. The RPD or relative standard deviation (RSD) for field duplicate samples (and unspiked compounds for MS/MSD samples) is calculated according to the following formulae:

Precision (for duplicate samples -- two data points):

$$\frac{D_1 - D_2}{[D_1 + D_2]/2} \times 100 = RPD$$

Where: D_1 = Larger Sample Value
D_2 = Smaller Sample Value (duplicate)
RPD = Relative Percent Difference

(for duplicate samples -- more than two data points):

$$\frac{SD}{AVE} \times 100 = RSD$$

Where: SD = Standard Deviation
AVE = Average (Mean Value)
RSD = Relative Standard Deviation

Standard deviation (SD) is calculated as follows:

$$SD = \sqrt{\frac{(y_i - y_m)_2}{(n-1)}}$$

Where: y_i = measured value of the $_i$th replicate
y_m = mean of the replicate measurements
n = number of replicate measurements

12.2 ACCURACY ASSESSMENT

To determine the accuracy of the analytical procedures, samples are selected at minimum specified frequencies and spiked with a known amount of the analyte or analytes to be evaluated. In general, one MS/MSD pair should be included in every set of 20 samples tested on each instrument and surrogate spike compounds should be included with each sample for organic analyses. The increase in concentration of the analyte, compared to the reported value of the same analyte in the unspiked sample, determines the percent recovery. Daily control charts are plotted for each commonly analyzed compound and are instrument-specific, matrix-specific, and analyte-specific. The percent recovery for a spiked sample is calculated according to the following formulae:

Accuracy (for analyte spike recoveries):

$$\frac{SSR - SR}{SA} \times 100 = \%\ R$$

Where: SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added
%R = Percent Recovery

Accuracy (for surrogate spike recoveries):

$$\frac{Cm}{Ca} \times 100 = \%\ R$$

Where: Cm = Concentration Measured
Ca = Concentration Added
%R = Percent Recovery

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\% \text{ Completeness} = \frac{D_t - D_u}{D_t} \times 100$$

Where D_t = Total number of data points
D_u = Total number of data points determined to be unusable/rejected

13.0 CORRECTIVE ACTION

The need for corrective action occurs when a circumstance arises that threatens the quality of the data output. For corrective action to be initiated, awareness of a problem must exist. In most instances, the personnel conducting the field work and the laboratory analyses are in the best position to recognize a problem or nonconformance that will affect data quality. Keen awareness on their part can frequently detect minor instrument changes, drifts, or malfunctions which can be corrected. If major problems arise, sampling and laboratory personnel are in the best position to determine the proper corrective action and initiate it immediately, thus minimizing data loss. Therefore, the field sampling and laboratory analysis personnel will have prime responsibility for recognizing a nonconformance and initiating a corrective action.

Each nonconformance shall be documented by the personnel identifying it or originating the call for corrective action. For this purpose, an audit report, an internal memorandum, or a letter may be used, as appropriate. Documentation shall include:

- An identification of the individual(s) identifying the nonconformance or originating the call for corrective action;
- A description of the nonconformance;
- Any required approval signatures;
- The method(s) for correcting the nonconformance (corrective action) or a description of the variance granted;
- A schedule for completing the corrective action.

A nonconformance report will document the corrective action. Samples that are affected will be listed on the nonconformance report.

Decisions on whether to take corrective action and the actions taken will be made by the OMC Project Manager. When a corrective action is taken by any of the field or analytical laboratory personnel, they will be responsible for notifying the Sampling Coordinator so that surveillance of the affected sampling or analysis system can be intensified in the field, if necessary.

Ultimately, the personnel performing the sampling and analysis procedures and results must participate in decisions to take corrective actions. To reach the proper decision, each individual must understand the program objectives and data quality necessary to meet these objectives.

DQOs for this program are presented in Section 3.3. All personnel involved in the analytical components of this project will receive a copy of this section and will be informed of these objectives. Each individual will be responsible for notifying the Project QA Manager whenever a measurement system is not generating data within these objectives.

If a situation arises requiring corrective action, the following closed-loop corrective action system will be used:

- Define the problem;
- Assign responsibility for investigation the problem;
- Investigate and determine the cause of the problem;
- Determine corrective action course to eliminate the problem;
- Assign responsibility for implementing the corrective action;
- Determine the effectiveness of the corrective action and implement the correction;
- Verify that the corrective action has eliminated the problem;
- If the corrective action is not completely successful, loop back to the first step.

The Project QA Manager will review the QA/QC data that are generated. Any excursions from the project objectives will be evaluated to determine the cause. Additional QA/QC samples may be collected and analyzed, or additional QA/QC tests may be identified and conducted to isolate the cause. Any corrective actions implemented will be documented to EPA Region V in the next quarterly report.

13.1 ANALYST CORRECTIVE ACTION

All analyst corrective actions are to be followed according to the recommendations in the Laboratory SOP for each method, as identified in Attachment 1. Specific corrective actions taken by laboratory analysts are also outlined in the Laboratory QA Manual.

13.2 QUALITY ASSURANCE CORRECTIVE ACTION

Any out-of-criteria performance discovered through routine auditing by the Laboratory QA Officer will be reported to the OMC Project Manager in writing. The affected results will then be reviewed, and a decision will be made concerning data applicability and/or corrective actions. Such decisions will be documented in writing and reported to EPA Region V for final decision.

Corrective action alternatives may include any of the following:

1. Qualification/flagging of affected data;

2. Discarding affected data and a review of the necessity of the data point discarded;

3. Discarding data and re-analysis;

4. Discarding data, re-sampling, and re-analysis.

13.3 FIELD CORRECTIVE ACTION

The Sampling Coordinator is responsible for all QA and corrective actions in the field. Any deviations from the QA protocols defined in the O&M QAPP must be justified, approved by the OMC Project Manager, and properly documented.

Any corrective actions that must be taken due to unacceptable field performance will be documented and approved by the OMC Project Manager prior to implementation. All field corrective actions will be noted on corrective action forms. These forms may be initiated by field sampling personnel, the Project QA Manager, or the OMC Project Manager. An example of a Corrective Action Request Form is provided on Figure 6.

14.0 QA REPORTS TO MANAGEMENT

Annual QA Reports will be prepared to inform management of the current project status. The QA Report will include the following:

1. Periodic assessment of the measurement data precision, accuracy, and completeness;
2. Results of any performance audits and/or system audits;
3. Significant QA/QC problems identified and recommended solutions;
4. Detailed references to O&M QAPP modifications.

Additionally, any incidents requiring corrective action will be fully documented. The Project QA Manager will prepare a report summary for the OMC Project Manager. The findings shall be factual, concise, and complete. Any required supporting information will be appended to the report.

A QA Report will be prepared annually and will be delivered to the OMC Project Manager.

14.1 QUALITY ASSURANCE PROJECT PLAN CHANGES

Any necessary changes to the O&M QAPP will be documented as an addendum to this document.

BCM ENGINEERS

TABLES

TABLE 1

TARGET PARAMETERS AND REPORTING LIMITS
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Sample Matrix	Analytical Parameter	Compound	SW-846 Preparation Method	SW-846 Analytical Method	Reporting Limit (RL) (ug/L)	Performance Criteria (ug/L)
Monitoring Wells (W1 - W12)	PCBs	Aroclor 1016	3510B	8082	1.0	(a)
		Aroclor 1221			1.0	(a)
		Aroclor 1232			1.0	(a)
		Aroclor 1242			1.0	(a)
		Aroclor 1248			1.0	(a)
		Aroclor 1254			1.0	(a)
		Aroclor 1260			1.0	(a)
Waste Water (Water Treatment Systems - Influent, Lead Carbon, Effluent)	PCBs	Aroclor 1016	3510B	8082	1.0	(b)
		Aroclor 1221			1.0	(b)
		Aroclor 1232			1.0	(b)
		Aroclor 1242			1.0	(b)
		Aroclor 1248			1.0	(b)
		Aroclor 1254			1.0	(b)
		Aroclor 1260			1.0	(b)

RL : Reporting Limit
ug/L : micrograms per liter
PCBs : Polychlorinated biphenyls

SW-846 Methods derived from the following:
"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, Third Edition, November 1990 and promulgated updates (including September 1997 Update III).

(a) : The performance criteria for each monitoring well is defined in the O&M Plan as 5 parts per billion (ppb, alternatively identified as ug/L) above background, and is reported as total PCBs.

(b) : Waste Water performance criteria for the 3 water treatment systems are defined in the O&M Plan.

TABLE 2

SAMPLE LOCATIONS AND FREQUENCIES
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Sample Matrix/ Location	Sampling Frequency (a)	Compounds Analyzed	Minimum Field QA/QC (b)	Laboratory QA/QC
Monitoring Wells (W1 - W12)	Semi-Annually	PCBs	10% field duplicates 5% equipment rinsate blanks 5% MS/MSD	MS/MSD, blanks and calibrations per SW-846 Method 8082
Waste Water Influent	Monthly	PCBs	10% field duplicates 5% field blanks 0% MS/MSD (c)	MS/MSD, blanks and calibrations per SW-846 Method 8082
Waste Water Lead Carbon	Monthly	PCBs	10% field duplicates 5% field blanks 0% MS/MSD (c)	MS/MSD, blanks and calibrations per SW-846 Method 8082
Waste Water Effluent	Monthly	PCBs	10% field duplicates 5% field blanks 0% MS/MSD (c)	MS/MSD, blanks and calibrations per SW-846 Method 8082

QA/QC : Quality assurance/quality control
MS/MSD : Matrix spike/matrix spike duplicate

(a) : Sampling frequency may be reduced or increased, as indicated in the O&M Plan. The frequencies identified herein represent the frequencies in effect as of December 1996.

(b) : Field QA/QC frequencies for monitoring wells (groundwater) are minimum frequencies per sampling event. Field QA/QC frequencies for waste water are minimum frequencies during the entire project (running totals).

(c) : Project-specific MS/MSD samples that are collected in the field are not required for the water treatment system samples to ensure data of sufficient quality to make proper project decisions regarding the data generated.

TABLE 3

DATA QUALITY OBJECTIVES
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Laboratory DQOs

Sample Matrix	Parameter	EPA Method	Compound	Precision (% RPD)	Accuracy (% R)
Monitoring Wells (W1 - W12)	PCBs	8082	Aroclor-1248 (spike)	+/- 31	34-116
			TCMX (surrogate)	-	25-140
			DCB (surrogate)	-	13-154
Waste Water (Water Treatment Systems)	PCBs	8082	Aroclor-1248 (spike)	+/- 31	34-116
			TCMX (surrogate)	-	25-140
			DCB (surrogate)	-	13-154

Field DQOs

Sample Matrix	Sample Type	Parameter	Criteria	Precision (% RPD)	Accuracy
Monitoring Wells (W1 - W12)	Field Duplicates	PCBs	All positive values	< 35	
	Blanks	PCBs	All compounds		< RL
Waste Water (Water Treatment Systems)	Field Duplicates	PCBs	All positive values	< 35	
	Blanks	PCBs	All compounds		< RL

The completeness DQO for all analyses is 95%.
Precision and accuracy DQO criteria are taken from laboratory-derived results, where appropriate.

DQO : Data quality objective
RPD : Relative percent difference
R : Recovery
PCBs Polychlorinated biphenyls
TCMX : Tetrachloro-meta-xylene
DCB : Decachlorobiphenyl
RL : Reporting Limit
EPA : Environmental Protection Agency
CLP : Contract Laboratory Program

TABLE 4

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
ANTICIPATED SAMPLING EVENT QUANTITIES
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Sample Matrix	Laboratory Parameter	Monitoring Samples	Field Duplicates	Equipment Rinsate/ Field Blanks	MS Samples	MSD Samples	Matrix Total
Monitoring Wells (W1 - W12)	PCBs	12	2	1	1	1	17
Waste Water* (I, LC, E)	PCBs	9	1	1	0	0	11

MS : Matrix Spike
MSD : Matrix Spike Duplicate
I : Influent
LC : Lead Carbon
E : Effluent

* : Assuming all 3 locations at all 3 water treatment systems are collected during a sampling event.
The frequency of groundwater duplicate, blank and MS/MSD QC samples are per sampling event.
The frequency of waste water duplicate and blank QC samples are based on a running total, not per sampling event.
Project-specific MS/MSD samples collected in the field are not required for water treatment system samples.

TABLE 5

CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Sample Matrix	Laboratory Parameter	Recommended Container	Preservation	Holding Time	Sampling Frequency (a)
Monitoring Wells (W1 - W12)	PCBs	2 x 1 L amber glass with Teflon-lined lid	Cool to 4 °C	14 days extraction/ 40 days analysis	Semi-Annual
Waste Water	PCBs	2 x 1 L amber glass with Teflon-lined lid	Cool to 4 °C	14 days extraction/ 40 days analysis	Monthly

(a) : Sampling frequency may be reduced or increased, as indicated in the O&M Plan. The frequencies identified herein represent the frequencies in effect as of December 1996.

TABLE 6

DATA VALIDATION CHECKLIST
OPERATION AND MAINTENANCE PLAN
WAUKEGAN HARBOR, ILLINOIS

Project # 00-8094-01

[ ] Yes [ ] No	1	Chain-of-Custody Documentation	
[ ] Yes [ ] No	2	Laboratory Chronicle/Case Narrative	Laboratory: _____
[ ] Yes [ ] No	3	Sample Results	
[ ] Yes [ ] No	4	Surrogate Recovery Summary	Lab Report Number: _____
[ ] Yes [ ] No	5	Reagent Blank Summary	
[ ] Yes [ ] No	6	Matrix Spike/Matrix Spike Duplicate Summary	Lab Report Date: _____
[ ] Yes [ ] No	7	Initial Calibration Summary	
[ ] Yes [ ] No	8	Continuing Calibration Summary	Validation Date: _____
[ ] Yes [ ] No	9	Sample Run Logs	
[ ] Yes [ ] No	10	Sample Chromatograms and Reports	Initials: _____
[ ] Yes [ ] No	11	Calibration Chromatograms and Reports	
[ ] Yes [ ] No	12	Reagent Blank Chromatograms and Reports	
[ ] Yes [ ] No	13	Matrix Spike/Matrix Spike Duplicate Chromatograms and Reports	

Matrix:	Compounds:	Other:	Other:
Soil ( )	PCBs (X)	_____ ( )	_____ ( )
Water (X)	VOCs ( )	_____ ( )	_____ ( )

[ ] This narrative covers the analysis of the following sample(s) in accordance with the Waukegan Harbor Superfund Site Operation and Maintence Plan _____

_____

[ ] The specified holding time for this analysis (14 days to extract/40 days to analyze) was met.

[ ] The specified holding time for this analysis was not met for sample(s): _____

[ ] Secondary dilutions are reported for sample(s): _____

[ ] Surrogate recoveries (except diluted samples) were within acceptable limits (50-150%).

[ ] Surrogate recoveries (except diluted samples) were not within acceptable limits for sample(s):

_____

[ ] Surrogates were not used in this analysis.

[ ] _____

[ ] Spike recoveries were within acceptable limits (50-150%).

[ ] Spike recoveries were not within acceptable limits for sample(s): _____

[ ] Interferences in the following samples exceeded the spike concentration and required a diluted analysis, which qualifies the recovery data: _____

[ ] The presence of target compounds in the following samples exceeded the spike concentration and required a diluted analysis, which qualifies the recovery data: _____

[ ] The method blank(s) did not contain any target compounds or interfering peaks at or above the reporting limits (1 ug/L).

[ ] The following method blank(s) contained the following compounds or interfering peaks at or above the reporting limits: _____

[ ] _____

[ ] Initial calibration criteria were met for this analysis.

[ ] Initial calibration criteria were not met for this analysis for the following: _____

[ ] Continuing calibration criteria were met for this analysis.

[ ] Continuing calibration criteria were not met for this analysis for the following: _____

[ ] _____

BCM ENGINEERS

FIGURES

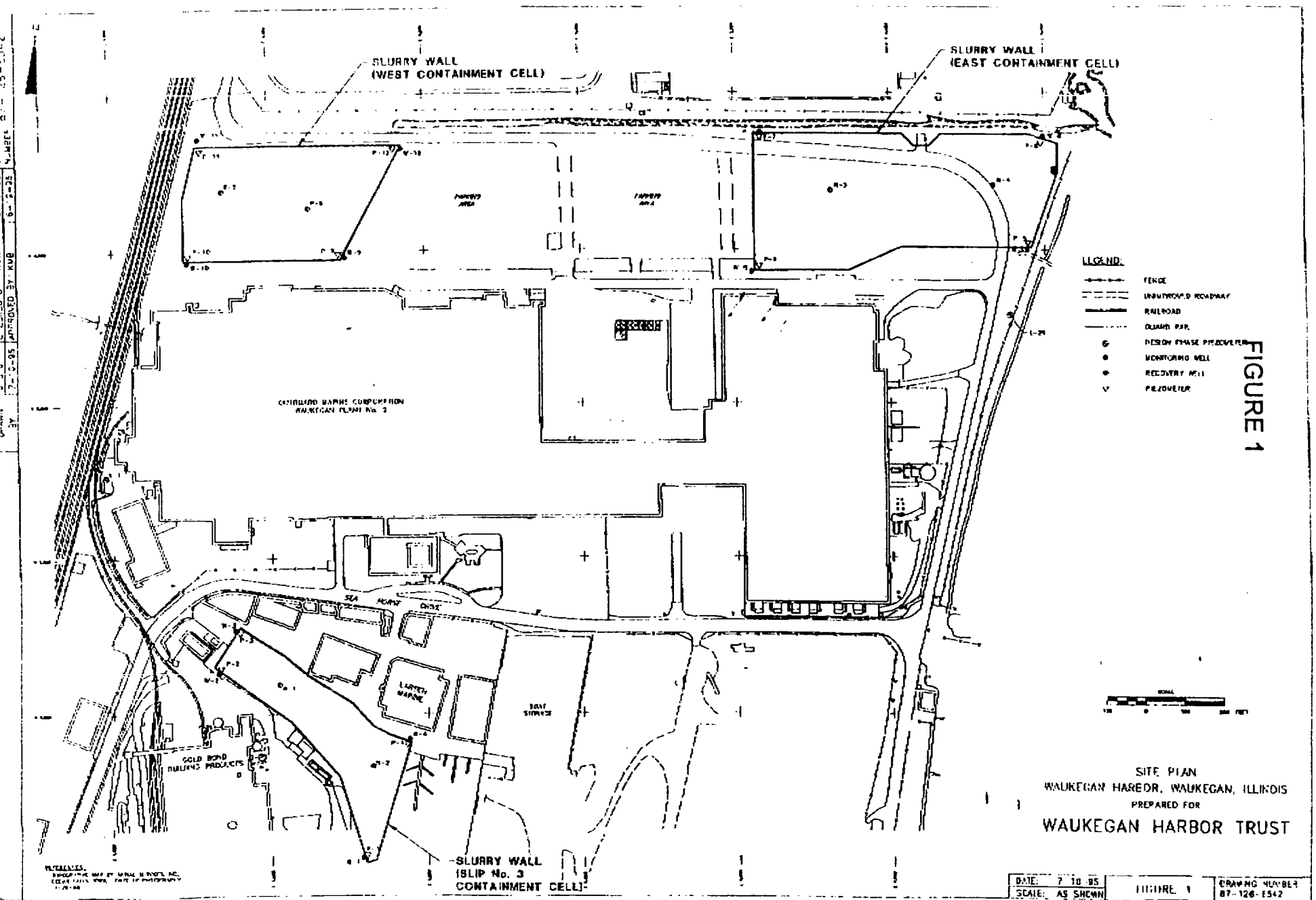


FIGURE 1

SITE PLAN
WAUKEGAN HARBOR, WAUKEGAN, ILLINOIS
PREPARED FOR
WAUKEGAN HARBOR TRUST

DATE: 7 10 95	FIGURE 1	DRAWING NUMBER
SCALE: AS SHOWN		87-126-E542

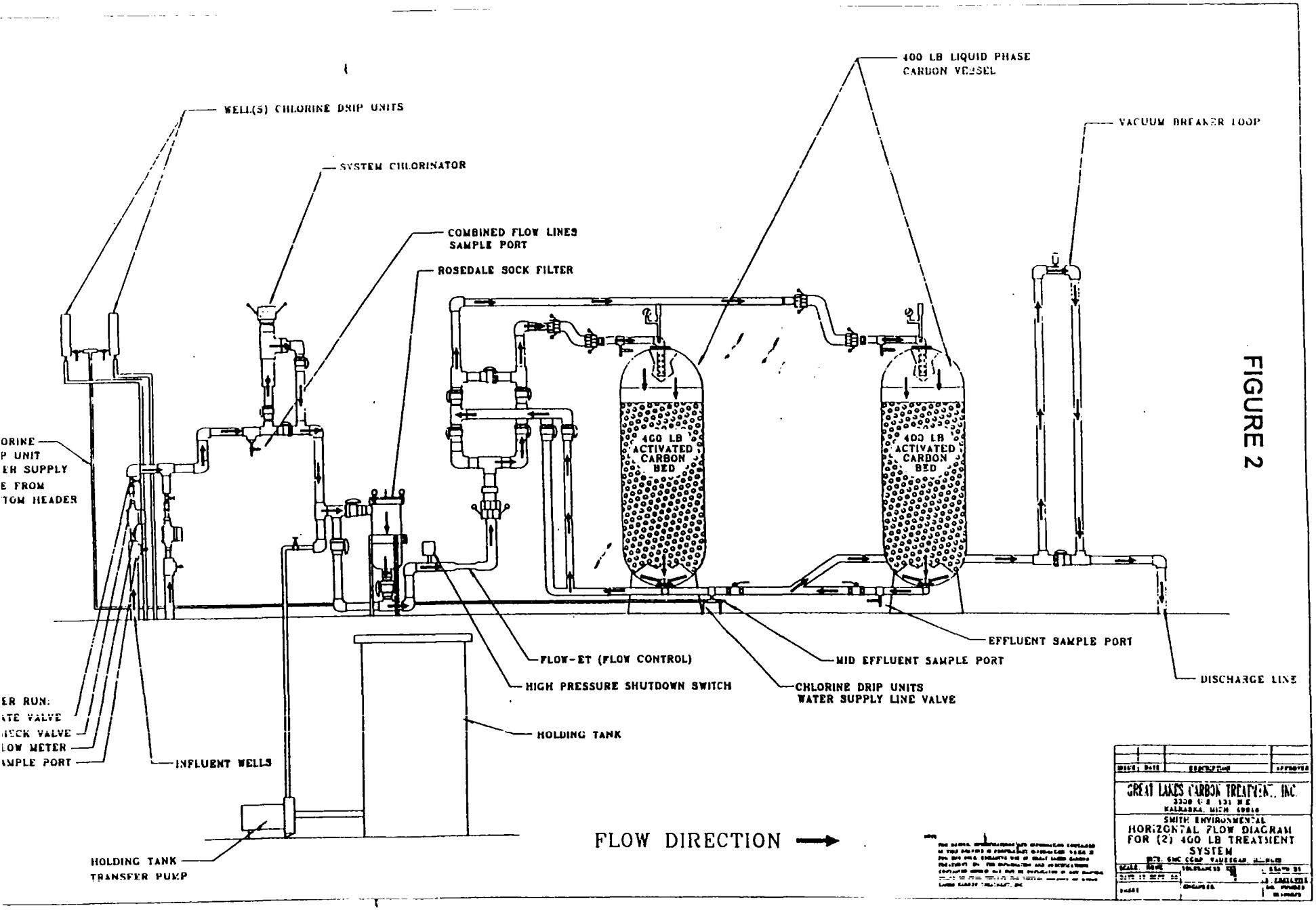

400 LB LIQUID PHASE CARBON VESSEL
WELL(S) CHLORINE DRIP UNITS
VACUUM BREAKER LOOP
SYSTEM CHLORINATOR
COMBINED FLOW LINES SAMPLE PORT
ROSEDALE SOCK FILTER
400 LB ACTIVATED CARBON BED
400 LB ACTIVATED CARBON BED
EFFLUENT SAMPLE PORT
MID EFFLUENT SAMPLE PORT
DISCHARGE LINE
FLOW-ET (FLOW CONTROL)
HIGH PRESSURE SHUTDOWN SWITCH
CHLORINE DRIP UNITS WATER SUPPLY LINE VALVE
HOLDING TANK
INFLUENT WELLS
HOLDING TANK TRANSFER PUMP
FLOW DIRECTION
GREAT LAKES CARBON TREATMENT, INC.
SMITH ENVIRONMENTAL
HORIZONTAL FLOW DIAGRAM FOR (2) 400 LB TREATMENT SYSTEM

FIGURE 2

FIGURE 3

GROUNDWATER MONITORING
FIELD DATA LOG
WAUKEGAN HARBOR SUPERFUND SITE
REMEDIAL ACTION/OPERATIONS & MAINTENANCE

DATE: ____________________

WELL #: ____________________ FIELD SAMPLERS: ____________________

DEPTH TO GROUNDWATER FROM TOC (FT): ________ DIAMETER OF WELL, d, (IN): 2 PAGE ___ OF ___

TOTAL WELL DEPTH FROM TOC (FT): ________ MINIMUM QUANTITY OF WATER TO BE PURGED:

HEIGHT OF WATER COLUMN IN WELL, W (FT) ________ Vw (GALS) WHERE Vw = 0.12*(d*d)*W: ________

PURGING EQUIPMENT USED: Suction Lift (Trash Pump)

SAMPLING EQUIPMENT USED: Peristaltic pump

TUBING TYPES:

In well/to pumps: Teflon lined polyethylene

In peristaltic pump: Silicon

WELL PURGING SUMMARY

TIME SAMPLED	PUMPING RATE (GPM)	TOTAL VOLUME (GAL)	TEMPERATURE DEGREES F	SPECIFIC CONDUCTANCE uS/cm	pH	TURBIDITY (ntu)	SAMPLE APPEARANCE & NOTES

TOTAL VOLUME PURGED: ________

ANALYTICAL SAMPLE COLLECTED

WEATHER: ____________________

COMMENTS: ____________________

NOTES: [illegible]

FIGURE 4

KEMRON

Client:_____

Sample Source:_____

Date:___/___/___ Time:_____

Taken By: _____

Parameters _____

_____

_____

Preservative _____

KEMRON
ENVIRONMENTAL SERVICES

№ 82878

CHAIN-OF-CUSTODY RECORD

Page _____ of _____

Project Contact:

Turn Around Requirements:

Project No.: | Project Name:

Sampler (print): | Signature:

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF SAMPLES	Hold	ADDITIONAL REQUIREMENTS

FIGURE 5

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Remarks:

*Homogenize all composite samples prior to analysis

White – Lab Yellow – Office Pink – Field

Outboard Marine Corporation	**General Corrective Action Request**	WAUKEGAN HARBOR SUPERFUND RA DATE:

NON-CONFORMING PROJECT / DEPT. AND ADDRESS

PROJECT/DEPT. REP. CONTACTED	CAR ORIGINATOR	TITLE

REQUIREMENT

SIGNED*: (Project/Dept. Rep) _____ SIGNED: (Auditor) _____
*Signature indicates understanding, not concurrence)

NONCONFORMANCE

SIGNED*: (Project/Dept. Rep) _____ SIGNED: (Auditor) _____
*Signature indicates understanding, not concurrence)

CORRECTIVE ACTION

DATE ACTION TO BE COMPLETED: _____
SIGNED: (Project/Dept. Rep) _____ DATE: _____

CORRECTIVE ACTION TAKEN TO PREVENT RECURRENCE

DATE ACTION TO BE COMPLETED: _____
SIGNED: (Project/Dept. Rep) _____ DATE: _____

FOLLOW-UP AND CLOSE-OUT PROPOSED FOLLOW-UP DATE: _____

CORRECTIVE ACTION VERIFIED BY: _____ DATE: _____

CORRECTIVE ACTION TO PREVENT RECURRENCE VERIFIED BY: _____ DATE: _____

DATE CAR CLOSED: _____ SIGNED (Auditor) _____

FIGURE 6

PAGE OF

Outboard Marine Corporation	General Corrective Action Request Supplement Form	WAUKEGAN HARBOR SUPERFUND RA
		DATE:

FIGURE 6

BCM ENGINEERS

ATTACHMENTS

BCM ENGINEERS

ATTACHMENT 1

LABORATORY STANDARD OPERATING PROCEDURES

BCM ENGINEERS

ATTACHMENT 2

FIELD SAMPLING STANDARD OPERATING PROCEDURES

KEMRON
ENVIRONMENTAL SERVICES

Document Control #:

STANDARD OPERATING PROCEDURE FOR ORGANIC ANALYSIS OF PCB'S METHOD 8082 SOP GCS10

Issue Date: 12 January 1999
Revision 2

Implementation Date: 12 February 1999

Last Review: 12 January 1999

KEMRON Environmental Services
109 Starlite Park
Marietta, Ohio 45750

Approved By:

Steve Learn, Semi-Volatile Supervisor — Date 1/13/99

Dennis S. Tebe, Laboratory Manager — Date 1-19-99

Daniel A. Musgrave, QA/QC Supervisor — Date 1/19/99

David L. Bumgarner, Director, Analytical Services — Date 1/20/99

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This method describes the procedures used to analyze PCBs in a variety of sample matrices. This method uses fused silica columns in order to separate target compounds. Table # 1 lists the compounds that may be determined by this method. This method follows the procedures outlined in SW-846 Update III Method 8082.

2.0 SAFETY PRECAUTION

Analysts using this method must be aware of certain hazards that are associated with performing the analysis.

2.1 Chemical Hazards

2.1.1 Several organic solvents are used for the preparation of standards and for the dilution of samples. These solvents include hexane, methylene chloride, methanol, benzene, and toluene. Each of these solvents are either carcinogenics, suspected carcinogenics or attack organs of the human body. Extreme care must be exercised when these compounds are being used. Gloves must be worn and proper ventilation must be in place before using any of the above mentioned solvents. All work with these solvents must be performed in a fume hood that has had the face velocity verified within the past year.

2.1.2 Concentrated organic standards are used to calibrate the instruments. Many of these chemicals have been found to cause cancer and must be handled with extreme care. Gloves must be worn while these compounds are being used. These compounds must be handled under a fume hood to prevent personnel exposure.

2.2 Thermal Hazards

Several zones on the GC are heated to high temperatures. Care must be exercised when working around these areas to avoid severe burns to the skin.

2.3 Broken Glassware Hazards

All dilutions that require the use of glassware must be made with care to reduce the possibility of cuts from broken glass. All broken or defective glassware must be disposed of in the broken glass container located in the laboratory.

2.4 Radioactive Hazards

Electron capture detectors are a sealed source radioactive ionization detector. Because it is a sealed source, the radioactive Ni 63 is not exposed to the environment and therefore poses little danger to the analyst. Wipe tests are performed on each detector on a semi-annual basis to check for Ni 63 contamination on the outside of the detector housing. These samples are sent away to an independent laboratory where they are analyzed for Ni 63 contamination. If detectors pass these wipe tests then the detectors will remain in service provided they are still operating within specifications. If the detectors have been leaking during the previous six months, then the detector must be removed from service immediately, and returned to Hewlett-Packard detector exchange program as soon as possible. Extreme care must be taken when handling a leaking detector to prevent personal exposure to radioactive materials.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Sample size requirements are determined by the matrix type of the sample.

3.1.1 Water samples require a volume of three (3) liters per sample. This will ensure the extraction laboratory has sufficient sample for the extraction of a matrix spike, and a matrix spike duplicate for the extraction batch. This amount will also allow for the sample to be re-extracted should the need arise.

3.1.2 Soil extractions require at least 90 grams of sample. This will ensure the extraction laboratory has sufficient sample for the extraction of a matrix spike, and matrix spike duplicate for the extraction batch. This amount will also allow for the sample to be re-extracted should the need arise.

3.2 Sample collection should be performed according to the outline set forth in SW-846. This will give the laboratory a sample representative of the sampling site.

4.0 METHOD PERFORMANCE

4.1 The detection limits for this analysis are subject to large changes caused by the sample matrix. Waters and soils that are free of co-extracted artifacts can yield very low detection limits while those that contain moderate to large amounts of co-extracted artifacts may have detection limits many times higher than the lowest achievable detection limits.

4.2 Table 1 gives a list of the compounds typically run under this method. The list also shows the method detection limits and reporting limits (RL) that can be expected in an analysis of soil or water samples that are free of co-extracted artifacts. The limits given in this table, represent the ideal matrix, and no guarantee is made to the client that these levels can be achieved. The limits reported to the client are those levels that can be achieved with that clients samples. The detection limit reported to the client in the data package will reflect the best detection limit that was able to be achieved in the analysis. Higher detection limits in the sample usually means that higher levels of co-extracted artifacts were found in the sample. Clean up of all extracts by Method 3620 are performed on all samples, method blanks, method QC, and matrix spikes. All extracts are treated in exactly the same manner.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 All possible measures are taken to eliminate interferences; however some samples have high levels of material that co-extract and make it virtually impossible to completely remove all of the interferences from the extracts. All samples, Blanks, LCS, matrix spikes, and matrix spike duplicates in a given batch undergo identical clean-up procedures. Any blank that contains target compounds at a level greater than the reporting limit will be cause for the entire batch of samples associated with the blank to be re-extracted. Any target compound found in the original extraction batch may be reported and flagged as estimated concentration only. Quality control criteria can be found in Table #2.

6.0 EQUIPMENT AND SUPPLIES

6.1 HP 5890 GC equipped with dual ECDs and capillary port injectors.

6.2 J&W DB-608 Wide bore capillary column 30 m x 0.53 mm I.D. and a 0.83 µm film thickness or equivalent.

6.3 J&W DB-5 wide bore capillary column 30 m x 0.53 mm I.D. and a 1.0 µm film thickness or equivalent.

6.4 HP 7673 dual tower autosampler

6.5 HP 3365 Chemstation analytical software.

6.6 IBM compatible PC with a 486 or higher micro-processor, hard drive storage, and QIC tape driven back-up capabilities.

6.7 Volumetric Flasks: 10, 25, 50 and 100 ml.

7.0 STANDARDS AND REAGENTS

7.1 Solvents: Hexane, Pesticide grade or higher quality

7.2 Surrogate Stock Standard - A 20 ppm surrogate stock standard of tetrachloro-m-xylene and decachlorobiphenyl is prepared from Supelco standard # 4-8460 or equivalent. This ampule contains 1.0 ml of 200 ppm of each of the two surrogate compounds. Exactly 1.0 ml of this standard is transferred to a 10 ml volumetric flask containing approximately 8 ml of pesticide grade hexane. Dilute the stock to the mark with pesticide grade hexane and invert three times to ensure proper mixing. The stock surrogate standard is now ready for use to prepare daily analytical standards. Secondary surrogate stock standards are prepared in an identical manner to that described above except that the high level source is Accustandard # CLP-032-R-WL-5x or equivalent.

7.3 PCB Stock Standards - Each of the PCB stock solutions are prepared in an identical manner. Each PCB high level solutions are purchased from Supelco at a concentration of 1000 ppm in iso-octane. From these high level ampules exactly 1.0 ml is transferred to a 100 ml volumetric flask containing approximately 80 ml of pesticide grade hexane. The Supelco standard numbers are:

Ar1016	4-8097 or equivalent
Ar1221	4-8098 or equivalent
Ar1232	4-4805 or equivalent
Ar1242	4-4806 or equivalent
Ar1248	4-4807 or equivalent
Ar1254	4-4808 or equivalent
Ar1260	4-4809 or equivalent

AR1016 and AR1260 are combined and analyzed together. Surrogate compounds are added by transferring 2.5 ml of stock surrogate standard prepared above to the volumetric flask. The flask is filled to the mark with pesticide grade hexane and inverted three times to ensure proper mixing. This yields standards having PCB concentrations at 10.0 ppm and surrogate concentrations at 0.50 ppm. The intermediate standard is now ready for use to prepare daily calibration standards. Secondary check standards stocks are prepared in an identical manner except that the high level source purchased from Accustandard or an equivalent source. All PCB standards are prepared individually and all contain the method surrogates. All PCB standards are stored at $\leq -10^{\circ}$ C and disposed of 6 months after the original date of preparation.

7.4 Calibration Standards:

PCB's - The PCB calibration standards are prepared from the working stock at the following levels:

Std # 1 - 50 ppb
Std # 2 - 100 ppb
Std # 3 - 250 ppb
Std # 4 - 500 ppb
Std # 5 - 1000 ppb
Std # 6 - 2000 ppb (optional)

These are made up by dilution on the working stock standard using the following scheme:

#1 - 20 x on the 1000 ppb #5 (950 µL hexane: 50 µL #5)
#2 - 10 x on the 1000 ppb #5 (900 µL hexane: 100 µL #5)
#3 - 4 x on the 1000 ppb #5 (750 µL hexane: 250 µL stock)
#4 - 20 x on the stock solution (10.0 ppm) (950 µL hexane: 50 µL stock)
#5 - 10 x on the stock solution (10.0 ppm) (900 µL hexane: 100 µL stock)
#6 - 5 x on the stock solution (10.0 ppm) (800 µL hexane: 200 µL stock)

8.0 CALIBRATION PROCEDURE

8.1 Gas Chromatograph Conditions

Columns -	J&W DB-608 Front
	J&W DB-5 Rear
Temp Prog -	160° C for 1.0 min
	5° C/min to 230° C for 3 min
	then 6° C/min to 280 for 6 min
Carrier Gas -	Helium at 7 ml/min
Ionizing Gas -	Ar/Me @ 60 ml/min
ECD Temp -	350° C
Injector Temp -	225° C
Injection volume -	2.0 ul

8.2 Run the following series of calibration standards after a hexane blank used to assess system cleanliness:

50 ppb Arochlor 1016/1260
100 ppb Arochlor 1016/1260
250 ppb Arochlor 1016/1260
500 ppb Arochlor 1016/1260
1000 ppb Arochlor 1016/1260
2000 ppb Arochlor 1016/1260 (optional)

500 ppb Arochlor 1016/1260 (second source)
500 ppb Arochlor 1221
500 ppb Arochlor 1232
500 ppb Arochlor 1242
500 ppb Arochlor 1248
500 ppb Arochlor 1254

500 ppb Arochlor 1221 (second source)
500 ppb Arochlor 1232 (second source)
500 ppb Arochlor 1242 (second source)
500 ppb Arochlor 1248 (second source)
500 ppb Arochlor 1254 (second source)

In AFCEE cases, a five point calibration is used for each positively identified Arochlor.

8.2.1 This sequence must be run whenever any major changes are made to the instrument. This curve must have a %RSD [standard deviation/average calibration factor (Cf)] of less than or equal to 20 %. If the % RSD is greater than 20 the analyst may quantitate samples by plotting against the 5 point curve using linear regression ($y = mx + b$) provided the coefficient of correlation is 0.995 or greater. If the coefficient of correlation is less than 0.995, then a new curve must be run.

8.2.2 The sequence of the sample run then proceeds as follows:

10 samples
Arochlor 1016/1260 check standard @ 500 ppb
10 samples etc.

8.2.3 The check standards must either be less than 15% difference using the formula (Ci-Cc/Ci)100 where Ci = initial calibration factor and Cc = check standard calibration factor, or yield a check standard concentration that is within 15% of the expected value using the formula: (Cc-Ca/Cc)100 where Cc is the expected concentration of the check standard and Ca is the

concentration calculated using the calibration curve. Inability to achieve %D of less than 15 when running a set of continuing calibration check standards will indicate a problem and will necessitate instrument maintenance.

8.2.4 A standard containing a mixture of Arochlor 1016 and Arochlor 1260 will include many of the peaks represented in the other five Arochlor mixtures. Such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Arochlors. Standards of the other five Arochlors are necessary for pattern recognition. These standards are also used to determine single point calibration factors for each Arochlor, assuming that the Arochlor 1016/1260 mixture demonstrates the linearity of the detector.

8.3 Running Samples and Check Standards

8.3.1 Assuming that the GC system is in good working order then the samples may be run as described above. If a check standard fails to yield Cf's with % difference of ± 15 % then a second check standard may be analyzed. Upon failure of two consecutive check standards a new curve must be run and the samples, bracketed within the bad check standard, must be re-run.

8.4 Blanks, LCS, Matrix Spikes, and Matrix Spike Duplicates

8.4.1 With each extraction batch of samples (maximum of 20 actual samples per batch) a blank, a LCS (blank fortified with target compounds), a sample fortified with target compounds (matrix spike), and a spike duplicate of this sample also fortified with target compounds (matrix spike duplicate) must be extracted and run along with the other samples in the batch. For AFCEE work, the MS and MSD count as actual samples. All extracts in a batch will undergo identical cleanup. The spike level is added to the sample at a concentration at the mid-point of the calibration. Water samples are spiked at 2.5 ug/L. Soil samples are spiked at 83.3 ug Kg. The MS MSD samples may be waived if insufficient sample is available.

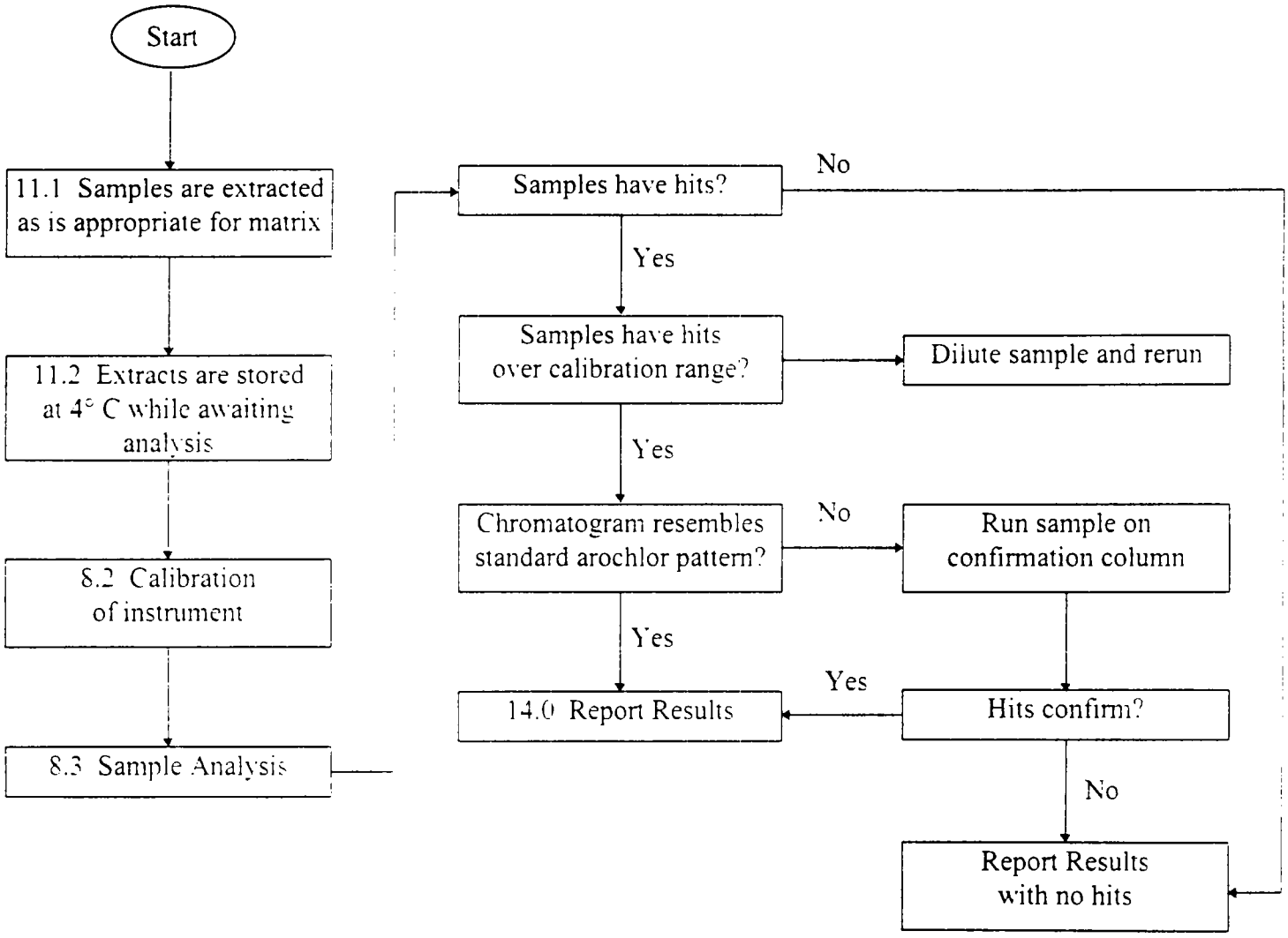
9.0 SAMPLE PREPARATIONS

9.1 All samples submitted to the laboratory for analysis under this method must first be extracted before the analysis can begin. Methods found in SW-846 detail extraction procedures suitable for a wide variety of sample matrices.

9.2 Soil samples submitted for analysis must undergo solvent extraction by sonication. This procedure is detailed in SOP EXPO2. This SOP is found under separate cover.

9.3 Water samples submitted for analysis must undergo solvent extraction by separatory funnel. This procedure is detailed in SOP EXPO1. This SOP is found under separate cover.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 All samples are extracted by the appropriate technique.

11.2 After the samples have been extracted, they must be stored at 4° C ± 2° C until the instrumental analysis can proceed. The samples must be analyzed within 40 days from the day of the extraction.

11.3 The instrument(s) are calibrated according to section 8.0 of this document.

11.4 Continuing check standards must be run for Arochlors 1016 and 1260 after a series of 10 samples have been run (or every 12 hours, whichever is more frequent) to ensure the instrument response has not changed. The percent difference in the RF for each compound of interest must be less than 15 percent for the quantitation column and less than 15 percent for confirmation of a tentatively identified compound. Second column confirmation is performed when requested in the client contract, or when matrix effects are present. In order to report a sample containing less than the PQL for a particular target compound, the sample must run behind a check standard having a % difference of less than 15.

11.5 A compound will be considered tentatively identified if a peak is found with the calculated daily retention time windows. The retention time windows are calculated by analyzing a standard over a 72 hour period, and calculating the standard deviation of each of the target compounds' retention time and multiplying each of these standard deviations by 3. This ± value is the retention time window for that compound. The mean absolute retention time used for defining windows will be taken from the continuing calibration standards. Multiresponse compounds such as Aroclors will have retention time windows calculated. However the identification of these compounds is based primarily on pattern recognition using 3 - 5 peaks of the multiresponse compound. This pattern recognition serves as confirmation of the Arochlor unless otherwise specified by client request.

11.6 If the calibration factors of the check standards are within operational limits, ±15 % difference of the mean ICAL calibration factors, ten (10) additional samples may be analyzed. If the check standards fail to meet the percent difference criteria, a second check may be analyzed. Upon failure of two consecutive check standards, a new five point calibration curve must be run. All samples must be bracketed by check standards which pass method criteria of ± 15 % difference when compared to the initial curve.

11.7 The raw data is processed using the chem station software and the target compound concentrations are entered in the LIMS report sheets.

11.8 The data is reviewed two times by the data review team; once for completeness and a second time for data validation.

11.9 The data goes to the data entry team where it is entered into the LIMS system followed by a third review of the completed data package.

11.10 Integration and quantitation of PCBs can be performed in a variety of ways. The goal is to arrive at a sample result that accurately reflects the Aroclor concentration in the sample. The method used by this laboratory shall be to integrate the entire Aroclor using a baseline which connects each valley of each congener peak with the valley of the next peak. Since the calibration standards have all been integrated exactly the same way, the quantitations not only are accurate but the baseline variations that can be caused by matrix interfering components are not assimilated into the quantitation and therefore will not alter the concentration of the sample by introducing extraneous area into the integration. Each major peak selected for quantitation will be treated as a single component. Each peak must pass all calibration criteria listed in section 8.2.1 and section 8.3.1 of this document. Quantitation of an unknown sample will be the average of the concentrations of each of the major peaks selected. A major peak is one that is at least 25% of the largest peak in the Aroclor.

12.0 DETAILS OF CALCULATION

12.1 Calculation of standard deviation the standard deviation is calculated using the formula:

$$S = \left(\frac{\sum (x_i - \bar{x})^2}{n-1} \right)^{1/2}$$

where:

x_i = the ith measurement of the variable x
$\bar{x}$ = the average value of x
n = the number of replicates

12.2 Calculation of % difference or drift.

The % difference or drift is calculated using the formula:

$$\%\,D = \left(\frac{C_t - C_m}{C_t}\right) x100$$

where:

C_t = the true value of the check standard
C_m = the measured value of the check standard

12.3 Calculation of % RSD

The % RSD is calculated using the formula:

$$\%\,RSD = \frac{s}{\bar{x}}\ x100$$

where:

s = the standard deviation
$\bar{x}$ = the average value of x

12.4 Calculation of % Recovery

$$\%\,R = \left(\frac{C_S - C_R}{C_T}\right) x\ 100$$

where:

C_S = the concentration found in the spiked sample
C_R = the concentration found in the reference sample
C_T = the true value spiked into the sample

12.5 Calculation of RPD

$$RPD = \frac{(x_1 - x_2)}{(x_1 + x_2/2)}\ x\ 100$$

where:

x_1 = the concentration of the sample
x_2 = the concentration of the sample duplicate

13.0 QUALITY CONTROL (QC) REQUIREMENTS

13.1 A batch is defined as a group of samples which are extracted together. A batch may contain a maximum of 20 samples. With each batch of samples extracted, a laboratory control sample (LCS) and a method blank must also be extracted. It is recommended that at least one sample for the batch be extracted three times. The last two extractions should be fortified with a spiking solution to provide a matrix spike, and matrix spike duplicate (MS & MSD). All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch. A standard containing a mixture of Arochlor 1016 and Arochlor 1260 is used as the spike in the LCS, MS, and MSD. The LCS, MS and MSD will all be spiked so as to yield a concentration that falls within the mid point of the curve.

13.2 After a new five point initial calibration curve is run on the instrument, a midpoint standard from a secondary source must be analyzed to ensure that the calibration standards were made properly, and to monitor the calibration standard to determine when the standard must be disposed of. The standard must agree within 15% in order for the curve to be considered acceptable.

13.3 The LCS must have recoveries for each compound that falls into the ranges given in Table #1. Failure to fall within these limits will require the entire batch to be re-extracted and re-analyzed for the analytes that failed to meet these criteria.

13.4 The method blank cannot contain amounts of any target analytes which are over the reporting limits (RL). If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. Any results between the MDL and RL will be reported, and the data appropriately qualified.

13.5 In order to monitor the extraction efficiency in each sample, a surrogate solution containing 2,4,5,6-tetrachloro-m-xylene and decachlorobiphenyl is added to each sample in the extraction batch. Since surrogate limits vary depending on the protocol, consult the QAP associated with the particular project. The recoveries for at least one of these surrogates must fall within the limits of Method 8000A recovery. If any individual sample has both surrogates outside the given limits, then the sample must be re-extracted. For AFCEE projects, only decachlorobiphenyl is required to fall within the set limits.

13.6 Compounds are identified by their peak retention time on the GC. Retention time windows are needed to evaluate samples in order to determine if a given peak represents an actual pesticide. To calculate the window the analyst is required to perform statistical operations on chromatograms from over a 72-hour period. The retention time window is calculated by multiplying the standard deviation of each peak by 3.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

14.1.1 The reporting requirements depend upon the need of the client. KEMRON offers four levels of data reporting which are described in some detail below. Prior to data entry into the LIMS, (either manual or automatic), all data must undergo thorough review in the department. This review will consist of 100% review by the primary analyst. This must be followed by a 100% review of each data package by another qualified analyst. The section supervisor or qualified designee must review each data package for completeness and adherance to Kemron analytical policies. The supervisor or qualified designee may conduct the 100% review of the data packages while reviewing for completeness and adherance to Kemron analytical policies provided s/he signs off on the review document in all applicable locations.

14.1.2 Level 1 reporting provides the client with the results for all samples Submitted for analysis. No other documents or raw data are provided with this level of report.

14.1.3 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.1.4 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.1.5 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

14.2 Reporting Requirements

14.2.1 An aroclor is identified by comparison of the chromatographic pattern with the pattern of a standard of the suspected aroclor.

14.2.2 The aroclor component peaks must fall with the calculated retention time window. If coelution or interfering components prohibits accurate assignment of the aroclor, another arochlor component may be used provided the calibration criteria are re-assessed using the new component.

15.0 PREVENTIVE MAINTENANCE

15.1 In order to minimize the downtime of the instrumentation, preventive maintenance is performed on a routine basis. The injection port liners and septa are changed regularly. Additionally, from time to time when the peak shape of the standards in the chromatograph is deformed, the front portion of the analytical column is clipped to improve performance. The ECD's are changed out when the calibration of the instruments becomes increasingly difficult.

16.0 REFERENCES

16.1 U.S. EPA 40 CFR Part 136. "Guidelines Establishing Test Procedures for the Analysis of Pollutant Under the Clean Water Act: Final Rule and Interim Final Rule and Proposed Rule.

16.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* SW-846, 3rd Edition. December 1996. Update III Method 8082.

TABLE # 1
KEMRON'S QA OBJECTIVES AND ANALYTICAL METHODS FOR PCB ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
PCB-1016	12674-11-2	8082	39-113	0-46	0.29	0.50
PCB-1221	11104-28-2	8082	15-175	0-38	0.32	0.50
PCB-1232	11141-16-5	8082	15-210	0-35	0.41	0.50
PCB-1242	53469-21-9	8082	35-154	0-35	0.35	0.50
PCB-1248	12672-29-6	8082	34-116	0-31	0.36	0.50
PCB-1254	11097-69-1	8082	25-135	0-36	0.30	0.50
PCB-1260	11096-82-5	8082	44-129	0-41	0.15	0.50

* Values are statistically derived from laboratory control samples and are updated annually. Actual control limits may vary.

TABLE # 1 (continued)
KEMRON'S QA OBJECTIVES AND ANALYTICAL METHODS FOR PCB ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	MDL SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
PCB-1016	12674-11-2	8082	10-241	0-49	7.7	17
PCB-1221	11104-28-2	8082	10-180	0-39	11	17
PCB-1232	11141-16-5	8082	11-205	0-36	14	17
PCB-1242	53469-21-9	8082	30-165	0-37	12	17
PCB-1248	12672-29-6	8082	31-175	0-39	11	17
PCB-1254	11097-69-1	8082	22-149	0-37	4.4	17
PCB-1260	11096-82-5	8082	10-272	0-62	9.1	17

* Values are statistically derived from laboratory control samples and are updated annually. Actual control limits may vary.

Compound Name	Wipe Reporting Limits ug/wipe
Ar1016	1.0
Ar1221	1.0
Ar1232	1.0
Ar1242	1.0
Ar1248	1.0
Ar1254	1.0
Ar1260	1.0

Detection limits are determined by statistical analysis of 7 replicates of spiked (reagent) waters. MDL = 3.14 (Standard Deviation) of the 7 replicates

TABLE # 2
Quality Control Criteria
PCBs
Method 8082

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Initially and upon failure of two consecutive continuing check samples	Ave Cf < 20% RSD	Evaluate and use linear or higher order calibration equation, recalibrate instrument (1)
Continuing calibration	Every ten samples (including method blanks and QC)	≤ 15% difference	Rerun CCV once more, upon 2nd failure recalibrate instrument
Method Blank	One per batch (20 samples maximum per batch)	Target compounds < reporting limits or < MDL x 2 (2)	Re-extract entire batch associated with blank Qualify data and address in narrative
Laboratory Control Sample (LCS)	One per batch (20 samples maximum per batch)	Within designated ranges (3)	Investigate, evaluate, repeat batch if necessary or qualify data and address in narrative (5)
Matrix Spike	One per batch (20 samples maximum per batch)	Within designated ranges (4)	Qualify data and address in narrative
Matrix Spike Duplicate	One per batch (20 samples maximum per batch)	Within designated ranges (4)	Qualify data and address in narrative
Surrogate Compound	Surrogate is placed in each sample, blank, and QC	Within designated ranges	Repeat analysis or qualify data and address in narrative

(1) Evaluation criteria is often project specific, generally ≤ 0.99 is a sufficient co-efficient of correlation
(2) Acceptance criteria are analyte specific
(3) Control limits set at three standard deviations from the mean recovery
(4) Advisory limits only; same as for LCS
(5) The decision to re-analyze or to qualify data is based on the number and severity of the outliers

TABLE # 3
KEMRON ENVIRONMENTAL SERVICES
Semivolatile GC Laboratory Maintenance / Runlog

Date ____ Column ID ____ Analyst ____ SOP # GCS09 Rev. # 0 ____ 8081A
Instrument ID ____ Data Subdirectory ____ SOP # GCS10 Rev. # 1 ____ 8082
SOP # GCS01 Rev. # 0 ____ 8100 SOP # GCS02 Rev. # 0 ____ 8015B Mod (DRO) SOP # GCS04 Rev. # 2 ____ 8151
SOP # GCS03 Rev. # 0 ____ PRO SOP # GCS02 Rev. # 0 ____ 8015B Mod (Alcohol) SOP # GCS07 Rev. # 0 ____ 8011

Daily Check	
gases >500 psi	
change liner	
change septum	
Preventative Maintenance	
change o-ring	
clip column (____ cm)	
injection port seal (goldseal)	
change gases	

Additional Maintenance

Problem: ____

Action Taken: ____

Returned to Control? Yes ____ No ____ Additional Discussion on Page ____

Run #	Sample Description	Dilution Factor	Method File	Analysis Complete	Comments

Comments:

√ = Compliant Analysis, Reanalysis complete
RR = Rerun
RR# = Rerun at a specified level

SMI = Sample matrix interference
MT = Missed Tune, beyound method tuning requirements
RE = Re-extraction analysis

Reviewed By: ____

IS or SS = Interference with internal and/or surrogate standards
CC = Continuing calibration failed

KEMRON ENVIRONMENTAL SERVICES

Figure 1
SV - GC

Date: _____
Analyst: _____
Method: _____
Instrument: _____
Work Group: _____

	Analyst
System Performance Check	
Initial Calibration	
Average RF	
Linear Reg or Higher Order Curve	
Second Source Standard % Difference	
Check Standards	
Project / Client Specific Requirements	
Special Standards	
Blanks	
TCL's	
Surrogates	
LCS (Laboratory Control Sample)	
Recoveries	
Surrogates	
MS / MSD / Duplicates	
Samples	
TCL Hits	
Surrogates	
Calculations & Correct Factors	
Dilutions Run	
Reruns	
Check Sample Histories	
Ending Check Standards	
Excel Spreadsheets	
Case Narrative	
Corrective Action	
Results Reporting / Data Qualifiers	
WorkGroups Traceability	
Client Data Package Assembly	
Check for Completeness	

Primary Reviewer Initials & Date Checked

_____

Secondary Reviewer Initials & Date Checked

_____

_____ • *Check for compliance with Method and project-specific requirements*
_____ • Check the completeness of the reported information
_____ • Check the information for the report narrative
_____ • Check the reasonableness of results

Supervisory Review Initials & Date Checked _____

Comments: _____

_____

NA - Not Applicable

DL - Diluted Out

√ - Checked & OK

KEMRON
ENVIRONMENTAL SERVICES

Document Control #:

STANDARD OPERATING PROCEDURE FOR SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTIONS FOR PESTICIDES AND PCB'S SOP EXP01 SW846 Method 3510C, 3500B, and CFR Method 608

Issue Date: 4 February 1998
Revision 7

Implementation Date: 4 March 1998

Last Review: 4 February 1998

KEMRON Environmental Services
109 Starlite Park
Marietta, Ohio 45750

Approved By:

Chad E. Barnes, Extraction Supervisor — Date: 02/06/98

Dennis S. Tepe, Assistant Laboratory Manager — Date: 02/09/98

Daniel A. Musgrave, QA/QC Supervisor — Date: 2/9/98

David L. Bumgarner, Director, Analytical Services — Date: 2/9/98

Table of Contents

1.0 SCOPE AND APPLICATION

This method describes a separatory funnel procedure for extracting and isolating pesticides and PCB's from aqueous samples. The method also describes the concentration and cleanup procedures involved in preparing the extract for analysis by GC. This method follows the procedures outlined in SW-846 Method 3510C, 3500B and 40 CFR Chapter 1 (7-1-95 Edition)

2.0 SAFETY PRECAUTIONS

2.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical compound should be treated as a potential health hazard.

2.2 Proper lab coat, gloves, and safety glasses should be worn while performing this extraction.

2.3 **CAUTION:** If using a regular 2 liter separatory funnel instead of a vent-separatory, initial venting of methylene chloride should be done immediately after the separatory funnel has been sealed because methylene chloride creates excessive pressure and should be done periodically while shaking the funnel.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in 1 liter glass containers with teflon lined caps.

3.2 Sample volume needed for analysis is 1 liter but if sample volume is less than 1 liter, record the actual volume of sample and then dilute sample to 1 liter with deionized water to perform extraction. If sample is significantly less than 1 liter, notify the TSR.

3.3 Sample preservation should be at, 4°C, and the sample maximum holding time from date of collection is seven (7) days.

4.0 METHOD PERFORMANCE

Not Applicable

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 Interferences may be caused by contaminants in solvents, reagents, glassware and other sample preparation.

5.2 Emulsions after a shake may occur in which the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical methods. If the emulsion can not be broken (recovery of less than 80 % of the methylene chloride) transfer sample, solvent, and emulsion into extraction chamber of a continuous extractor and proceed with a continuous extraction method.

5.3 Other interferences that may be encountered are discussed in method 3500B SW-846.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

6.1.1 Zymark TurboVap II concentration workstation

6.1.2 Glas-Col 3D Floor Shaker or equivalent

6.1.3 Nitrogen Evaporator: Meyer N-Evap Analytical Evaporator or equivalent

6.1.4 Solid Phase Extraction (SPE) Manifold

6.2 Apparatus or equipment

6.2.1 Kuderna-Danish (K-D) apparatus:

6.2.1.1 Concentrator tube: 15 ml graduated (Supelco 6-4684M or equivalent)

6.2.1.2 Evaporation Flask: 50 ml attached to a concentrator tube with a clip or spring

6.2.1.3 Snyder column: Three ball macro

6.2.2 Water bath: Heated, with concentric ring cover, capable of temperature control (±5° C). The bath should be used in a hood.

6.3 Glassware

6.3.1 2 liter separatory funnel with teflon stopcock or an automatic 2 liter venting separatory funnel with teflon stopcock.

6.3.2 Graduated cylinder: 1 liter

6.3.3 250 ml ground glass erlenmeyer or flask

6.3.4 Zymark 200 ml concentrator tubes with 1.0 ml endpoint

6.3.5 Calibrated 40 ml vial

6.4 Other Supplies

6.4.1 Syringes - various sizes

6.4.2 pH paper: Wide range (0-13).

6.4.3 Glass wool.

6.4.4 Stainless steel funnel or glass funnel

6.4.5 Autosampler vials: 2 ml capacity

6.4.6 Boiling chips: 10/40 mesh

6.4.7 Florisil column: 1000 mg (J.T. Baker 7213-07 or equivalent)

7.0 REAGENTS

7.1 Deionized water.

7.2 Sodium hydroxide solution 10 N: Dissolve fourty (40) grams NaOH in deionized water and dilute to 100 mL.

7.3 Sodium sulfate: Granular, anhydrous.

7.4 Methylene chloride: Pesticide grade or equivalent.

7.5 Hexane: Pesticide grade or equivalent.

7.6 Sulfuric acid solution (1:1): Slowly add 50 mL of sulfuric acid to 50 mL of deionized water.

7.7 Ethyl ether: Pesticide grade or equivalent.

7.8 Pesticide surrogate (Supelco 4-8460) mix: Methanol solution containing:

Decachlorobiphenyl	1.0 mg/mL
2,4,5,6-tetrachloro-m-xylene	1.0 mg/mL

7.9 PCB Spike - Aroclor 1660 (Mix of Aroclor 1016 and 1260) 25 ug/mL

7.10 Pesticide spike (Supelco 4-8913) mix: Methanol solution containing:

Heptachlor	2.0 mg/mL
Aldrin:	2.0 mg/mL
Dieldrin	2.0 mg/mL
Endrin	2.0 mg/mL
4,4'-DDT	2.0 mg/mL
4,4'-DDD	2.0 mg/mL
4,4'-DDE	2.0 mg/mL
Alpha-BHC	2.0 mg/mL
Beta-BHC	2.0 mg/mL
Gamma-BHC	2.0 mg/mL
Delta-BHC	2.0 mg/mL
Endosulfan I	2.0 mg/mL
Endosulfan II	2.0 mg/mL
Endosulfan sulfate	2.0 mg/mL
Endrin aldehyde	2.0 mg/mL
Heptachlor epoxide	2.0 mg/mL
Endrin ketone	2.0 mg/mL
Methoxychlor	2.0 mg/mL

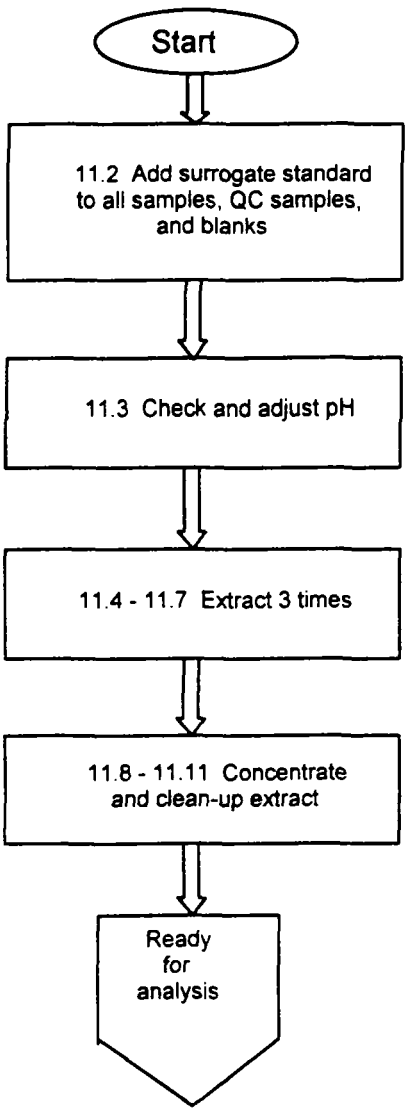
8.0 CALIBRATION PROCEDURES

Not Applicable

9.0 SAMPLE PREPARATIONS

Extraction method 3510C and 3500B from SW-846

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 STEP-BY-STEP ANALYTICAL PROCEDURES

11.1 Rinse all extraction glassware with methylene chloride.

11.2 Use a 1-liter graduated cylinder, measure 1 liter of sample and transfer it to a separatory funnel. Add 200 ul of the pesticide surrogate to all samples, spikes and blanks. Add 250 ul of the pesticide spike solution to the LCS (Laboratory Control Sample) and to samples designated as matrix spikes. (Add 100 uL of the PCB spike solution to the LCS (Laboratory Control Sample) and to samples designated as matrix spikes for PCB only analysis). Record sample volume and all surrogate and spike additions.

11.3 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to between five (5) and nine (9) using 10N sodium hydroxide solution or sulfuric acid (1:1).

11.4 Add 60 mL of methylene chloride to the separatory funnel.

11.5 Seal the funnel with a teflon stopper and shake the separatory funnel vigorously for two (2) minutes with periodic venting to release excess pressure. **NOTE:** Methylene chloride creates excessive pressure rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. If using the automatic venting separatory funnel, screw teflon cap on and turn separatory funnel upside down. Place the stopcock in the venting position and place funnel on 3-D shaker. Once funnel is secured in place, shake for 2 minutes.

11.6 Allow the layers to separate for at least ten (10) minutes. The methylene chloride layer will be on the bottom. Drain the methylene chloride layer through a funnel plugged with glass wool and filled with about 10-20 g of granular sodium sulfate into a 250-mL Erlenmeyer flask.

11.7 Repeat the extraction (11.4-11.6) two (2) more times using 60 mL of fresh methylene chloride each time and combine the extracts in a 250-mL Erlenmeyer flask.

11.8 Concentration of extracts using the Kuderna-Danish apparatus.

11.8.1 Attach a 15-mL concentrator tube to a 500-mL Kuderna-Danish (K-D) flask with a spring and a clip.

11.8.2 Transfer the extract to the K-D flask and rinse the Erlenmeyer flask with 10-20 mL of methylene chloride and add it to the K-D flask.

11.8.3 Add a boiling chip to the K-D flask and attach the Snyder column to the top. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80° - 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. When the apparent volume reaches 1 mL, remove the K-D apparatus from the water bath and allow it to cool for at least ten (10) minutes.

11.8.4 Add 50 mL of hexane through the Snyder column into the K-D flask and concentrate the extract as described in 11.8.3, raising the temperature of the water bath to 90° - 95° C.

11.8.5 Adjust the final volume to 10 mL by either adding hexane or concentrating it with the nitrogen evaporator (N-evap). To use the N-evap, turn on the heater to 35° C. Remove the concentrator tube from the K-D apparatus and place it in the N-evap. Turn on the gas tank and lower the needle so that it is about a centimeter above the liquid. Remove the concentrator tube from the N-evap when the apparent volume is about 9 mL. Rinse the sides of the tube with hexane and adjust the volume to 10 mL. Record the final volume.

11.9 Concentration of extracts using the Turbovap II.

11.9.1 Rinse the concentration (200 ml) tubes with methylene chloride.

11.9.2 Transfer the extract into the tube, rinse the erlenmeyer and add it also to the tube.

11.9.3 Place the tube into the Turbovap II which is set at 45° C and close the cover.

11.9.4 Press the sensor button on the Turbovap and start the concentration process for each cell by pushing the start/stop button for each cell.

11.9.5 Adjust the gas flow to get a nice helical flow that does not spit out the sample.

11.9.6 When the endpoint is reached, the light next to its start/stop button will blink and the beeper sounds briefly every thirty seconds.

11.9.7 Remove the tube from the Turbovap and add 50 ml of hexane making sure that a proper exchange has happened and place the tube back into the Turbovap and concentrate the extract as described in 11.9.1 - 11.9.6.

11.9.8 When the endpoint is reached, remove the tube from the Turbovap and transfer the extract into a calibrated 40 ml vial.

11.9.9 Adjust the final volume to 10 ml by adding hexane. Record the final volume.

11.10 Florisil Cleanup (for PCB only analysis skip to 11.11)

11.10.1 Prepare the florisil column by pre-eluting the column with 5 mL of hexane. Discard the hexane.

11.10.2 Just prior to the exposure of the florisil to air, transfer 5 mL of extract to the florisil column and collect the extract in a 40 mL pre-cleaned VOA vial calibrated to 5 mL.

11.10.3 After the sample has gone through the florisil, elute the column with 1 mL of 94:6 hexane: ether followed by 3 mL of 1:1 hexane: ether followed by 1 mL of 94:6 hexane: ether. Collect these elutions in the VOA vial.

11.10.4 Concentrate the volume to 5 mL using the N-evap as in step 11.8.5.

11.10.5 Transfer the extract to an autosampler vial and seal, mark sample level and label it with sample ID, extraction date and test.

11.11 Acid/Florisil Cleanup (**for PCB's only**)

11.11.1 Add 5 mL of concentrated sulfuric acid to the extract.

11.11.2 Stopper the tube and shake well for one (1) minute, venting once or twice to release any pressure. Allow the layers to separate for ten (10) minutes.

11.11.3 Pipet the cleaned up extract off the top and repeat if necessary. Pipet off 5 mL of extract for florisil cleanup.

11.11.4 Prepare florisil column and add the extract as in step 11.10.

11.11.5 After the sample has gone through the florisil, elute the column with 3 mLs of 94:6 hexane: ether. Collect in the VOA vial.

11.11.6 Concentrate the volume to 5 mL using N-evap as in step 11.8.5.

11.11.7 Transfer the extract to an autosampler vial and seal, mark the sample level and label with sample ID, extraction date and test.

12.0 DETAILS OF CALCULATION

Not Applicable

13.0 QUALITY CONTROL (QC) REQUIREMENTS

13.1 A reagent blank, quality control sample, matrix spike and matrix spike duplicate are extracted with each batch.

13.2 All reagent blanks, quality control samples, matrix spikes and matrix spike duplicate are subjected to exactly the same analytical procedures as those used on actual samples.

13.3 Quality Control Corrective Action is stated in KEMRON Environmental Services Laboratory Quality Assurance Plan Section 13.

14.0 DATA REPORTING REQUIREMENTS

Not Applicable

15.0 PREVENTATIVE MAINTENANCE

Not Applicable

16.0 REFERENCES

16.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition. Update I, II, IIA, and III Method 3510C, 3500B, 3665A, and 3620B.

16.2 CFR Chapter 1 (7-1-95 Edition) Method 608

BCM ENGINEERS

ATTACHMENT 3

FIELD ANALYTICAL STANDARD OPERATING PROCEDURES

GROUNDWATER SAMPLING

1.0 SCOPE AND APPLICATION

This method s applicable to collecting representative groundwater samples from monitoring wells, recovery wells, or piezometers at the OMC site, using pumps that are dedicated to the project and tubing that is dedicated to each well.

2.0 SUMMARY OF METHOD

Groundwater monitoring well samples are collected using dedicated lines made of Teflon®-lined polyethylene tubing which have been properly decontaminated. The well is evacuated with a trash (suction-lift type) pump or peristaltic pump to remove stagnant water from the casing and to allow infiltration to recharge the well with fresh formation water. After the well is sufficiently evacuated, either a trash pump or peristaltic pump is used and the sample is immediately collected and distributed into appropriate sample containers for chemical analysis.

3.0 SPECIAL CONSIDERATIONS

- This technique is limited to wells which utilize pumps and tubing.
- Coordinate bottle shipments for each parameter at each location with the Laboratory Project Manager and the quantities of quality control samples with the OMC Project Manager prior to each sampling event.

4.0 APPARATUS

Pumps for evacuation, evacuation tubing (Teflon®-lined polyethylene), electric or other reliable water level probe

5.0 PROCEDURE

5.1 Sampling Equipment Decontamination (Excluding Pumps and Tubing)

All sampling equipment, excluding the dedicated pumps and tubing, will be decontaminated prior to use according to the following procedure:

1 Non-phosphate detergent plus tap water wash
2 Tap water rinse
3 Distilled/deionized water rinse

4. Hexane rinse (all solvents pesticide grade or higher)*
5. Total air dry or pure nitrogen blow out
6. Distilled/deionized water rinse

Clean the electric water level probe with soap and deionized/distilled water.
* : Do not use hexane to decontaminate parts that may be degraded by the solvent (i.e., plastics, rubber, Teflon® tape, etc.).

5.2 Pump and Tubing Decontamination

If submersible pumps and tubing are ever used for well evacuation, they will be decontaminated in the following manner, if they are not dedicated to one location:

1. Detergent wash and tap water rinse, or steam cleaning, of outside pump casing, tubing, and cables.

2. A minimum 20-gallon flush of potable water through the pump. Pumps do not need to be cleaned between well locations. New polyethylene tubing is recommended for each well.

5.3 Sampling Preparation

To ensure that collected samples are truly representative of groundwater conditions, monitoring wells should not be sampled less than two weeks after development. This requirement may be waived on a case-by-case basis.

5.3.1 Static Water Level Measurement

Static water level in the well will be measured using an electric well probe (or by other reliable means, i.e., wetted tape) and tape measure to 0.01 foot accuracy to the surveyed reference point marked on the well casing. This measurement will be recorded on a Water Level Data Form and/or a Field Data Log Form.

5.3.2 Well Evacuation and Sampling

The well must be evacuated or purged prior to sampling to remove stagnant water in the casing, which may not be representative of aquifer conditions.

1. The static well volume will be calculated by multiplying the height of the column of water by the volume of water per foot. Evacuate a minimum of three volumes of standing water in the well. In low recovery wells (less than one gallon per minute yield), purging three volumes may not be practical. In this case, the well will be purged once to near dryness. For OMC monitoring wells, based on historical data it is appropriate to

discharge the water directly to the ground. This option should be evaluated by the Project Manager prior to sampling.

2. Measure temperature, specific conductance, pH, and turbidity (OMC SOP Nos. Harbor1-Harbor4, respectively) on each well volume until these parameters are found to be constant (defined as <10% change over two successive well volumes, or <10 NTU for turbidity). Record all measurements for each well volume in the field log. The measurements taken on the final well volume can be used as the measurements of record for that well.

3. Peristaltic pumps and dedicated one-half inch Teflon®-lined polyethylene tubing will be used. Decontamination of the pumps is not necessary, but non-dedicated portions of piping or tubing (tees and flow control valves) must be decontaminated after use in each well.

4. Purge rates will be adjusted, whenever possible, to allow purging of three times the well volume without causing excessive drawdown and loss of suction to the pump. The purge rate must be sufficiently slow to minimize the suspension of solids. Purging will be accomplished in one of the following ways:

   a. When the yield of the well is known, the pump rate will be adjusted to maintain a constant water level. The tubing should be placed above the well screen at a depth no greater than 6 feet below the static water level for the duration of the purge, if possible.

   b. If the recovery rate of the well is slower than one gallon per minute, the pump will be lowered to the bottom of the well from the start of purging to accommodate the drawdown that will occur.

5. If a new well is installed that recovers slowly, the well will be allowed to recover prior to sampling as follows:

   a. If the original water level covered the top of the well screen, the water level will be allowed to recover to a height sufficient to cover the screen.

   b. In high yielding wells, the water level will be allowed to recover to a least 80 percent of the original water height.

   c. In low yielding wells which require more than two hours to recover, the water level will be allowed to recover until a sufficient volume of water is available for the sample analysis. Sampling will then proceed according to the sampling priority list shown in Section 6.0.

6. If the appropriate pumps are not available, bailing may be used for evacuation, although it is not recommended. The bailer should be laboratory-cleaned Teflon or stainless steel.

7. Sample immediately after the completion of well evacuation recovery. The **only** exception to this is a well that has been pumped to near dryness that takes longer than two hours for the water level to recover to a height of water that will cover the screen. Slow recovery wells will be sampled as soon as they have recovered to an appropriate volume.

8. Record the sample appearance in the field log. Slowly and carefully transfer the sample into laboratory-prepared sample containers for only those parameters which are applicable, in the sequence outlined below:

   Volatile organic compounds (VOC)
   Total petroleum hydrocarbons (TPH)
   Polychlorinated biphenyls (PCB)
   Extractable organics
   Total metals
   Dissolved metals
   Turbidity

9. Fill 40-milliliter vials immediately, if VOCs are required, leaving no head space. Place the septum cap in the vial with the Teflon® side toward the sample.

   In case where preservation is required to extend volatile analytical holding times, preserve samples using 1:1 hydrochloric acid (HCl) to a pH less than 2.0. To determine the volume of 1:1 HCl, fill a test vial with water taken from the final purge volume. Add two to three (2-3) drops of 1:1 HCl using a new Pasteur pipette. Close the vial, invert the mix, then check with pH paper. *If the pH is greater than 2, add additional HCl. Record the* amount of acid needed to achieve a pH less than 2 in the field notebook. Discard the test sample and preserve the samples with the volume of HCl required to achieve a pH less than 2.

10. Turn the 40-mL vial on end and tap to ensure there is no air trapped below the vial cap. If air bubbles are present, discard and collect new sample.

11. Fill all other sample containers to the shoulder, leaving some head space for addition of preservative and expansion of the sample. Verify the quantities/types of bottles and preservatives for each analysis with the laboratory, if necessary

12. Filter samples for metals, if required.

13. Wipe the outside of the sample containers and label prior to shipment. Each label will contain the following information:

    - Well identification
    - Date and time sample taken
    - Sampler's name
    - Requested analysis
    - Indication of preservation

14. Place samples in a cool (4°C) environment under appropriate chain-of-custody procedures and ship to the laboratory as soon as possible.

6.0 DOCUMENTATION

At a minimum, the following information will be recorded for each well in a field log or on an OMC monitoring well data sheet (an example of a monitoring well data sheet is attached):

- Well identification
- Date, time, and weather conditions during sampling and purge
- Total depth of well (note the reference point found on the well casing)
- Depth of water (note the reference point found on the well casing)
- Purge volume (total)
- Purge rate
- pH, specific conductivity, temperature and turbidity of each well volume
- Signature of sampler
- Appearance of sample

All field records will be maintained as part of the project file. All notes will be recorded in ink and will be signed and dated by the field sampler.

7.0 REFERENCES

USEPA office of Emergency and Remedial Response, A Compendium of Superfund Field Operation Methods, EPA/540/P-87/001, December, 1987.

TREATMENT PLANT WASTE WATER SAMPLING

1.0 SCOPE AND APPLICATION

This method is applicable to ground, surface, and saline waters, as well as to domestic and industrial wastes.

2.0 PROCEDURE

2.1 While the treatment system is operating, collect influent, effluent, and lead carbon samples from the designated sample ports in each treatment system building.

2.2 Turn the handle on the sample port slowly to start the flow of water. Run the water for a few seconds to flush any potentially stagnant water from the sample port line.

2.3 Using pre-cleaned sample bottles received from the analytical laboratory, label the bottle with the sample identifier code, the date, the time, and the sampler's initials. Fill each bottle to at least the shoulder of the bottle. Try not to touch the inside of the bottle with the sample port spigot.

2.4 As soon as the bottle is filled, replace the sample cap tightly and wipe the exterior of the bottle.

2.5 Record the appropriate information in the treatment plant log and/or the field record book. Note which recovery wells are pumping at the time of sampling and whether influent samples are from single or combined flow influent ports.

2.6 Package samples for shipment following the procedures described in the QAPP.

3.0 FIELD LOGBOOK DOCUMENTATION

- Date and Time of each sample collection
- Description of samples taken
- Any other notable observations

WELL DEVELOPMENT OF GROUNDWATER MONITORING WELLS

1.0 SCOPE AND APPLICATION

This method is applicable to developing groundwater from monitoring wells using bailers.

2.0 SUMMARY OF METHOD

Groundwater monitoring wells are developed using PVC, Teflon, or stainless steel bailers which have been properly decontaminated. The well is evacuated to remove stagnant water from the casing, to allow infiltration to recharge the well with fresh formation water (see Section 5.3.2 for well purging requirements), and to create a surging effect to remove sediment which may have accumulated in the screen.

3.0 SPECIAL CONSIDERATIONS

3.1 The technique is limited to those wells which can accommodate a bailer, (i.e., wells without in-place plumbing and of sufficient diameter).

3.2 It is difficult, if not impossible, to evaluate a well's condition without specific information regarding construction details and well yield. If sufficient information is not available, attempts should be made through the Project Manager to obtain the necessary details.

4.0 APPARATUS

Pumps (submersible or surface) for evacuation
Evacuation tubing (polyethylene or Teflon-lined polyethylene)
Bailers (PVC, Teflon, or stainless steel)
Bailer leader, rope
Electric or other reliable water level probe

5.0 PROCEDURE

5.1 Well Development Equipment Decontamination

An optional steam-cleaning or high pressure water wash to remove residuals may be performed prior to the following steps. All sampling equipment will be decontaminated prior to use according to the following decontamination procedure:

1. Non-phosphate detergent plus tap water wash
2. Tap water rinse
3. Distilled/deionized water rinse
4. Total air dry or pure nitrogen blow out
5. Distilled/deionized water rinse

Whenever possible, use dedicated bailers and bailer leader. Clean bailer leader with soap and distilled/deionized water before use. Clean the electric water level probe with soap and distilled/deionized water.

5.2 Pump and Tubing Decontamination

Pumps and tubing used for well evacuation will be decontaminated in the following manner:

1. Detergent wash and tap water rinse or steam cleaning of outside pump casing/tubing, and cables.

2. 20-gallon flush of potable water through the pump. Surface pumps do not need to be cleaned between well locations. New or dedicated Teflon-lined polyethylene tubing is recommended for each well.

5.3 Sampling Preparation

To ensure that collected samples are truly representative of groundwater conditions, monitoring wells should not be sampled less than two weeks after development. This requirement may be waived on a case-by-case basis.

5.3.1 Static Water Level Measurement

Static water level in the well will be measured using an electric well probe (or by other reliable means, i.e., wetted tape) and tape measure to 0.01 foot accuracy to the surveyed reference point marked on the well casing. The volume of standing water contained in the well will be calculated prior to well purging by multiplying the height of the column of water by the volume of water per foot.

5.3.2 Well Evacuation

The well must be evacuated or purged prior to sampling to remove stagnant water in the casing which may not be representative of aquifer conditions.

1. Evacuate a minimum of ten volumes of standing water in the well. In low recovery wells (less than one gallon per minute yield), purging ten volumes may be difficult. In this case, the well will be purged several times to near dryness.

Generally, all evacuated water should be containerized for proper handling/treatment in the on-site treatment systems. However, in some cases, based on historical data, it is appropriate to discharge the water directly to the ground; this option should be evaluated by the Project Manager prior to sampling.

2. When requested, measure temperature, specific conductance, pH, and/or turbidity (OMC SOP Nos. Harbor 1, Harbor 2, Harbor 3, and Harbor 4) on each of the last several well volumes collected until these parameters are found to be constant (defined as <10% change in these parameters over two successive well volumes or less than 10 NTU for turbidity). Record all measurements in the field notebook or log form as they are measured. The measurements taken on the final well volume can be used as the measurements of record for that well.

6.0 DOCUMENTATION

At a minimum, the following information will be recorded for each well in a bound field notebook or on a Waukegan Harbor field data log:

- Well identification
- Date, time, and weather conditions during well development
- Total depth of well (note the reference point found on the well casing)
- Depth of water (note the reference point found on the well casing)
- Purge volume (total)
- Purge rate
- pH, specific conductivity, and temperature of last several well volumes
- Signature of field staff completing the well development

All field records will be maintained as part of the project file. All notes will be recorded in ink and will be signed and dated by the field staff.

7.0 REFERENCES

USEPA Office of Emergency and Remedial Response, A Compendium of Superfund Field Operation Methods, EPA/540/P-87/001, December, 1987.

USEPA Region II CERCLA Quality Assurance Manual, March 1988.

New Jersey Department of Environmental Protection, Bureau of Environmental Measurements and Quality Assurance, Field Sampling Procedures Manual, February 1988.

WATER LEVEL MEASUREMENT IN MONITORING WELLS, PIEZOMETERS, AND RECOVERY WELLS

1.0 SCOPE AND APPLICATION

This method is applicable monitoring wells, piezometers, and recovery wells.

2.0 APPARATUS AND MATERIALS

Water level tape, well keys

2.0 PROCEDURE

2.1 Clean the water level tape with soapy water, wash and deionized/distilled water rinse between each use.

2.2 For monitoring wells and piezometers:

2.2.1 Unlock and open the protective casing, and remove standpipe cap, if present.

2.2.2 Drop the water level tape into the standpipe until the electric indicator sounds.

2.2.3 Raise and lower the tape until you are sure the tape is just at the water surface, then measure the distance on the tape against the highest point of the standpipe, or the level mark on the standpipe, if present. (This is the point where the elevation of the pipe was surveyed).

2.2.4 Record the water level, and the date and time of the measurement, in the field log book and/or a water level data form (see attached examples). Repeat for each well and/or piezometer.

2.3 For recovery wells:

2.3.1 Unlock and remove the grating which covers the concrete casing, if present.

2.3.2 Insert the water level tape into the standpipe until the electric indicator sounds.

2.3.3 Raise and lower the tape until you are sure the tape is just at the water surface, then measure the distance on the tape against the highest point of the standpipe, or the level mark on the standpipe, if present. (This is the point where the elevation of the pipe was surveyed).

2.3.4 Record the water level, and the date and time of the measurement, in the field log book and/or a water level data form (see attached examples). Repeat for all three containment cells.

2.4 For measuring total depth in a well or piezometer:

2.4.1 Turn off the electronic detector and lower the tape into the water until it stops or goes slack.

2.4.2 Tighten up the slack so you can just feel the bottom.

2.4.3 Measure the distance on the tape against the highest point of the standpipe, or the level mark on the standpipe, if present. (This is the point where the elevation of the pipe was surveyed).

2.4.4 Record the total depth, and the date and time of the measurement, in the field log book and/or a water level data form. Note if the bottom feels soft or hard. This is an indicator of whether sediment is accumulating in the well and whether any maintenance or redevelopment is warranted.

3.0 FIELD LOGBOOK DOCUMENTATION

- Date and time of measurement
- Actual measurement of water level or total depth
- Any other notable observations

WASTE SAMPLING FROM DRUMS, STORAGE TANKS, OR CARBON VESSELS

1.0 SCOPE AND APPLICATION

This method is applicable to waste sampling from drums, storage tanks, or carbon vessels. Aqueous and solid sampling is discussed. These methods provide a representative sample of the container's contents.

2.0 SUMMARY OF METHOD

Discrete grab samples are collected from each container and transferred to sample bottles for laboratory analysis. Representative samples are acquired by inserting the sampling device on a diagonal from the container opening, collecting a cross section of its contents, where possible.

3.0 SPECIAL CONSIDERATIONS

- This method requires two people
- Health and safety issues must be carefully considered

4.0 SAMPLING EQUIPMENT AND DECONTAMINATION

4.1 Sampling Equipment

The following equipment may be used, depending upon the surveyed contents of the container:

Open tube sampler (liquid)
Hand auger/trowel or scoop and stainless steel bowl (solid)
Peristaltic pump (liquid, slurries)
Bacon-bomb sampler (sludge) or "sludge judge"
Gloves
Sample bottles (laboratory prepared)

4.2 *Sampling Equipment Decontamination*

Whenever possible, disposable sampling equipment will be used for container sampling. The equipment is left in the container to be disposed with the contents, or arrangements are made for disposal prior to sampling. When it is not possible to use disposable equipment, a field decontamination procedure will be followed. An optional steam cleaning or high pressure water wash to remove residuals may be performed before performing the following decontamination procedure:

1. Non-phosphate detergent plus tap water wash
2. Tap water rinse
3. Distilled/deionized water rinse
4. 10 percent nitric acid rinse* (trace metals or higher grade HNO_3 diluted with distilled/deionized water)
5. Distilled/deionized water rinse*
6. Hexane rinse** (pesticide grade or higher)
7. Total air dry or pure nitrogen blow out**
8. Distilled/deionized water rinse**

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* Only if sample is to be analyzed for metals
** Only if sample is to be analyzed for organics

4.3 Sample Containers

Sample bottles will be appropriately cleaned by the laboratory prior to being sent to the field. The sample containers will meet the volume and materials requirements of the analytical method to be performed. After the sample is placed in the container, the outside of the containers will be wiped clean with a cloth dampened with deionized water and then dried with a clean cloth.

5.0 SAMPLE COLLECTION PROCEDURE

5.1 Opening Storage Tanks or Carbon Vessels

5.1.1 Access ports on the top of the tanks/vessels will be used for sampling.

5.1.2 At least two people must always perform the sampling: one to open the hatch and/or collect the actual samples, and the other to observe, ready to assist or obtain emergency help if needed.

5.1.3 Prior to opening the hatch, the sampler should check the tank for a pressure gauge or relief valve. The manufacturer's instructions should also be reviewed and followed, if available.

5.1.4 The relief valve should be opened slowly to bring the tank to atmospheric pressure. If the tank pressure is too great or venting releases gases or vapors, discontinue venting immediately.

5.1.5 Monitor any release to the atmosphere with portable field instrumentation and record in field logbook.

5.1.6 If no relief valve exists, slowly loosen hatch cover bolts using a spark proof device to relieve pressure in the tank.

5.1.7 Do not remove hatch cover bolts until tank is at atmospheric pressure. Do not open tanks exhibiting high pressures, discontinue opening hatch cover if a release starts to occur.

5.2 Liquid and Slurry Sampling

Tanks/vessels containing liquids are sampled using an open tube sampler (thief) or peristaltic pump.

5.2.1 Open Tube Sampler

5.2.1.1 Insert the sampler into the center of the liquid to be sampled.

5.2.1.2 The sampler should be oriented on a diagonal from the point of entry, collecting a cross section of the tank contents.

5.2.1.3 Place gloved thumb securely over open end of tube and carefully withdraw the sample.

5.2.1.4 Retrieve the sampler and immediately transfer the sample into a sample bottle by removing gloved thumb. If applicable, fill VOA vials first.

5.2.1.5 If the sampling device is disposable, leave it in the tank sampled, unless storage tank is in active use. If in active use, place the device in an appropriate waste storage container.

5.2.1.6 If the sampling device is not disposable, decontaminate it thoroughly.

5.2.1.7 Sample bottles will be wiped down and then packaged and shipped to the laboratory.

5.2.2 Peristaltic Pump Sampling

The peristaltic pump will not be decontaminated. The only contact between the pump and the sample is the tubing, therefore, the tubing must be changed between sampling tanks to avoid contamination. The analysis of different parameters may require different types of tubing (e.g. silicone, viton, Teflon) to avoid contamination or reaction with the tubing. The peristaltic pump may be used to sample all liquid phases and slurries.

5.2.2.1 The end of the sample tube to be lowered into the fill pipe or hatch is weighted.

5.2.2.2 The tube is lowered to a depth of 6 inches, and the pump is turned on. At this point the tank contents is recorded, based on observation.

5.2.2.3 Continue step 5.2.2.2 until entire tank contents are surveyed. During these steps, the tank contents are containerized (drummed, etc.), but not actually sampled.

5.2.2.4 Mark the tubing once the entire tank is surveyed and record the depth of the tank.

5.2.2.5 Replace the tubing and sample the individual phases of the tank either separately or as a composite.

5.2.2.6 Retrieve the tubing and immediately transfer the sample into the sample container. If applicable, fill VOA vials first.

5.2.2.7 Sample bottles will be decontaminated and then packaged and shipped to the laboratory.

5.3 Solid Sampling

Samples should be collected as discrete grab samples. Depending on project objectives, compositing of samples in the field or laboratory may be acceptable. Soil samples are collected using a hand auger.

5.3.1 Insert the hand auger into the center of the exposed material to be sampled.

5.3.2 Using a hand auger, collect a core of material from a point diagonally opposite the point of entry.

5.3.3 If the solid is of shallow depth/thickness and can be safely reached by hand, a trowel may be used to collect several grabs or aliquots to obtain a representative sample of the overall material. These portions of material should be mixed thoroughly in a pre-cleaned stainless steel bowl. This compositing procedure shall not be used for VOA analyses.

5.3.4 Transfer the sample to a bottle using a trowel or scoop. If applicable, fill VOA vials first.

5.3.5 Decontaminate the sampling equipment thoroughly.

5.3.6 Sample bottles will be wiped down, packaged and shipped to the laboratory.

5.4 Sludge Sampling

Sludge samples will be collected using a Bacon-bomb (or similar) sampler or with a "sludge judge" (or similar). The bacon bomb may be used to sample a discrete layer or the bottom one inch of the container. A sludge judge may be used to collect a sample from a cross-section of the container. This will represent a composite of the material in the container. A sludge judge works in a similar manner to the open-tube sampler described in Section 5.2.1.

5.4.1 Lower the sampler carefully to the desired depth, allowing the line to the trigger to remain slack at all times.

5.4.2 When the desired depth is reached, pull the trigger line until taut.

5.4.3 Release the trigger line and carefully retrieve the sampler from the tank.

5.4.4 Unscrew the top of the sampler and transfer the sample to the clean sample container. If applicable, fill VOA vials first.

5.4.5 To sample the bottom 1-inch of a tank, lower the sampler carefully into the tank until it reaches the bottom of the tank.

5.4.6 As the sampler hits the bottom of the tank the sampler valve opens and fills.

5.4.7 As the sampler is raised, the valve closes.

5.4.8 Unscrew the top of the sampler and transfer the sample to the clean sample container. If applicable, fill VOA vials first.

5.4.9 Sample bottles will be decontaminated, packaged and shipped to the lab.

6.0 DOCUMENTATION

All notes will be recorded in ink in a bound field notebook and signed by the field sampler. At a minimum, the following information will be recorded for each container sampled:

- Container identification
- Date, time of sampling
- Matrix (solid, liquid)
- Sample description
- Method of sampling
- Any known information on container contents

7.0 REFERENCES

USEPA Office of Emergency and Remedial Response, A Compendium of Superfund Field Operation Methods, EPA/540/P-87/001, December 1987.

USEPA Region II CERCLA Quality Assurance Manual, March 1988.

ATTACHMENT 3

FIELD ANALYTICAL STANDARD OPERATING PROCEDURES

TEMPERATURE

1.0 SCOPE AND APPLICATION

This method is applicable to ground, surface, and saline waters, as well as to domestic and industrial wastes

2.0 SUMMARY

Temperature measurements may be made with any accurate, high quality mercury-filled thermometer or thermister with analog or digital read-out device.

3.0 SPECIAL CONSIDERATIONS

Ideally, temperature measurement devices shall be routinely checked against a National Institute of Standards and Technology certified thermometer, or equivalent. Alternatively for this project, the thermometer may be checked by immersing in a bath of distilled water and distilled ice that is at thermal equilibrium. The temperature reading of the thermometer must be between -0.5 and 0.5 degrees Centigrade (°C) or between 31 and 33 degrees Fahrenheit (°F) to be acceptable.

4.0 APPARATUS AND MATERIALS

Calibrated, mercury-filled thermometer

5.0 PROCEDURE

5.1 Use only a previously calibrated or checked mercury-filled thermometer.

5.2 Whenever it is removed from a field vehicle, allow the thermometer enough time to equilibrate to ambient temperature.

5.3 Insert the thermometer in situ when possible, or immediately in a grab sample.

5.4 Swirl the thermometer in the sample and take the temperature reading when the mercury column or read-out needle stops moving.

5.5 Record the temperature to the nearest 0.5 °C or 1.0 °F.

6.0 FIELD LOGBOOK DOCUMENTATION

- Instrument/thermometer used (currently utilizing a Hydac Conductivity, Temperature and pH Tester, Beta Technologies, Inc., Serial # 9508)
- Date
- Temperature range
- Sample temperature readings
- Identification of person performing temperature measurements

7.0 PRECISION AND ACCURACY

Precision and accuracy for this method have not been determined.

8.0 REFERENCES

APHA, AWWA, WPCF, Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 124, Method 212 (1980).

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, 170.2 (1979).

SPECIFIC CONDUCTANCE

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to ground, surface, and saline waters, as well as to domestic and industrial wastes.

2.0 SUMMARY

The specific conductance of a sample is measured by using a self-contained conductivity meter, Wheatstone Bridge-type, or equivalent.

3.0 SPECIAL CONSIDERATIONS

If the temperature of the sample is not at 25 degrees Celsius (°C) and the meter does not have automated temperature compensation, a compensation calculation is necessary (see Section 5.4).

4.0 APPARATUS AND MATERIALS

Hydac Conductivity, Temperature and pH Tester, Beta Technologies, Inc., Serial # 9508

5.0 PROCEDURE

5.1 INSTRUMENT SET-UP FOR THE HYDAC METER

5.1.1 Verify that the meter needle coincides with zero on the conductivity scale.

5.1.2 If necessary, adjust the meter zero by turning the bakelite screw on the meter face so that the needle coincides with the zero on the conductivity scale.

5.1.3 Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.

5.1.4 Plug the probe into the probe jack on the side of the instrument.

5.1.5 Check a 0.010 normal (N) potassium chloride (KCl) solution at 25.0 degrees Centigrade (°C). The standard reference solution should measure 1413 micromhos per centimeter (µmhos/cm) at 25.0 °C. Document the result in the field logbook.

5.1.6 Thoroughly rinse the probe with distilled, deionized water.

5.1.7 Put the probe in the solution to be measured.

5.2 TEMPERATURE

5.2.1 Set the MODE control to TEMPERATURE.

5.2.2 Allow time for the probe temperature to come to equilibrium with that of the sample before reading.

5.2.3 Read the temperature on the bottom scale of the meter in °C or in °F.

5.2.4 Record this reading in the field notebook.

5.3 CONDUCTIVITY

5.3.1 Switch the MODE control to the X100 scale. If the reading is below 50 on the 0-500 range switch to the X10 scale. If the reading is still below 50, switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in µmhos/cm. Measurements are not temperature compensated. If the temperature of the sample is not 25°C, make temperature correction in accordance with the appropriate calculation in Section 5.5 to convert the reading to 25°C.

5.3.2 When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should read within 2 percent of the original reading; if it is greater, the probe may be fouled and the measurement is in error. The probe must be serviced by a qualified technician before continued use.

NOTE: The CELL TEST does not function on the X1 scale.

5.4 CALCULATION

5.5.1 Utilize the following equation for temperature compensation when the sample temperature is not 25°C:

$$\text{Spec. Cond.}_{\text{(calculated)}} = \frac{M}{[1 + 0.0191\,(t-25)]}$$

where: M = measured value and t = sample temperature

5.5.2 Report results as Specific Conductance, µmhos/cm at 25°C. Report the results to three significant figures, or to the nearest 1 µmhos/cm.

6.0 FIELD LOGBOOK DOCUMENTATION

- Instrument (ID or serial number)
- Date of field calibration (REDLINE check)
- Temperature readings
- Salinity readings
- Salinity readings with CELL TEST button depressed
- Conductivity readings
- Conductivity reading with CELL TEST button depressed
- Temperature compensation calculations if sample temperature is not 25°C
- Identification of person performing calibration and sample measurements

7.0 PRECISION AND ACCURACY

The conductivity meter should have an accuracy of ± 2 percent of the true value. With satisfactory equipment, results within 1 percent of the true value can be obtained.

8.0 REFERENCES

APHA, AWWA, WPCF, Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 205 (1985); 18th Edition, Method 2510 (1992).

Annual Book of ASTM Standards, Part 31, "Water," Standard D1125-64, p. 120 (1976).

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, 120.1 (1979).

TURBIDITY

1.0 SCOPE AND APPLICATION

This method is applicable to ground, surface, and saline waters, as well as to domestic and industrial wastes.

2.0 SUMMARY

Turbidity in water is caused by suspended matter, such as clay, finely divided organic and inorganic matter, soluble colored organic compounds, and plankton and other microscopic organisms. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines through the sample. A turbidimeter is used for measrement, consisting of a nephelometer, a light source for illuminating the sample, and one or more photoelectric detectors with a readout device, with a sensitivity of 0.02 Nephelometric Turbidity Units (NTU) or less and a range of 0 to 40 NTU, at a minimum.

3.0 SPECIAL CONSIDERATIONS

- Determine turbidity on the day the sample is taken.
- Do not store for over 24 hours because irreversible changes may occur.
- Vigorously shake all samples before examination.

4.0 APPARATUS AND MATERIALS

4.1 Portable Turbidimeter, HF scientific, inc., Model DRT-15CE.

4.2 Sample tubes, clear colorless glass, scrupulously clean, inside and out.

4.3 Turbidity-free water, such as commercially bottled demineralized water.

4.4 Commercially available turbidity standards (such as 10.0 and 40.0 NTU).

5.0 PROCEDURE

5.1 The HF DRT-15CE turbidimeter is factory calibrated. Standardize the meter following the manufacturer's operating instructions (see the attachment to this SOP). The critical standardization steps are included herein

5.2 Standardize the instrument by placing the Reference Standard (0.02 NTU) in the optical well, switch to the " 10" range, indexing the cuvette by rotating until the position of the lowest reading is located, then adjusting the Reference Adjust in the appropriate direction to cause the display to read 0.02 NTU. The instrument is now ready to use on any range.

5.3 Check the accuracy of the instrument by measuring at least one freshly prepared standard (such as 10.0 or 40.0 NTU) on the range that will be used to measure the samples. Document the measurement check standard reading in a field log book.

5.4 Measure each sample by filling a clean tube to within approximately one-half inch of the top. Place the cap on the tube, carefully clean the outside surface with a lint-free wipe, place the sample in the optical well, and take the NTU reading directly from the display. Select the appropriate range for optimum readability.

5.5 Record the turbidity with two significant figures, or to the nearest 0.05 NTU.

6.0 FIELD LOGBOOK DOCUMENTATION

- Instrument used (currently utilizing a HF scientific, inc., turbidimeter, Model # DRT-15CE)
- Date
- Turbidity Reference Standard reading and check standard reading(s)
- Turbidity range(s) used
- Sample turbidity readings
- Identification of person performing turbidity measurements

7.0 PRECISION AND ACCURACY

Precision and accuracy for this method have not been determined.

8.0 REFERENCES

APHA, AWWA, WPCF, Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 133, Method 214 (1985).

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, 180.1 (1979).

0.02 NTU
HF Certified Reference Standard
*EPA Approved
Pure Turbidity Free Water
DO NOT OPEN*

Catalog No. 60047
The NTU value of the HF scientific, Inc. Reference Standard is 0.02 NTU. This value has been determined based on an EPA method for producing turbidity free water. HF scientific, Inc. certifies that this 0.02 NTU standard, is a pure water standard manufactured to meet or exceed the EPA requirements for turbidity free water. The value of the water has now been defined by EPA, therefore we can state with certainty that this standard, when used according to instructions, is 0.02 NTU.

Revised 12/31/92

*EPA approval means this standard meets or exceeds the performance criteria as specified in the United States Standard Method 2130 B.1

Reference Standard Indexing
For all HF Turbidimeters except the Micro-T

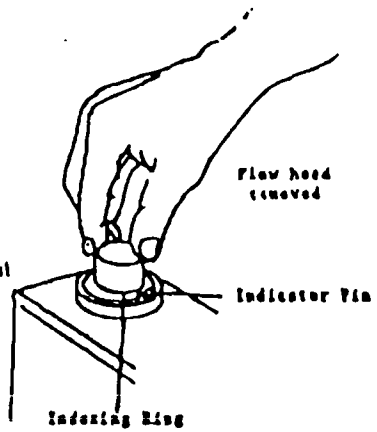
The EPA recommends that cuvettes used for instrument calibration, standardization or sample measurement be indexed. For quick and repeatable indexing of the reference standard, an indexing ring and locator pin are included with this reference standard.

The white locator pin may already be installed in the collar ring around the optical well of your turbidimeter.

To index your reference standard, slowly rotate the reference standard, at least one complete revolution, while observing the reading, and locate the position of the lowest reading. Without moving the reference standard, install the indexing ring over the ridged cap of the reference standard such that the notch on the ring aligns with the locator pin.

When standardizing this instrument in the future, simply insert the reference standard and rotate it until the notch on the indexing ring faces the locator pin. Standardize as per the instruction manual for your instrument. Please note that this reference standard is only indexed to the turbidimeter for which it was aligned.

Please refer to the instruction manual supplied with your instrument for instructions on standardizing and reference standard care.



DRT-15CE
PORTABLE TURBIDIMETER
OPERATING & MAINTENANCE
MANUAL

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FOREWORD

HF turbidimeters are manufactured to meet design criteria for nephelometers as described in Standard Methods For Examination of Water and Wastewater. HF turbidimeters are approved by the U.S. EPA* as a means to measure the turbidity of potable water, waste water, and other liquids.

HF turbidimeters provide a linear display of turbidity, throughout all ranges, in Nephelometric Turbidity Units (NTU). HF turbidimeters use solid state electronic components because they resist thermal variation and are not affected by normal line voltage fluctuations.

HF turbidimeters can be calibrated using HF scientific factory certified Secondary Standards or Formazin. Factory calibration is accomplished using HF scientific Secondary Standards, which are factory certified traceable to formazin, therefore, this instruction manual describes the proper procedures for calibration of HF turbidimeters using Secondary Standards.

HF turbidimeters use Formazin as the primary standard for calibration. Therefore, this instruction manual describes the proper procedures for calibration of HF turbidimeters using Formazin standards.

HF turbidimeter manuals are designed to assist the user in taking full advantage of the instrument in a majority of its applications. However, in the event that unusual circumstances or problems, not covered by this manual, arise please feel free to contact our local distributor or the manufacturer.

HF scientific, inc.
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Our engineering staff is available to help you with your specific needs.

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Figures

DRT-15CE (3/95)

I. SPECIFICATIONS FOR DRT-15CE

Ranges NTU	3 Ranges: 0-10, 0-100, 0 - 1000 NTU
Linearity	(+ or -) 1% /10, 5%/100, 10%/1000 of full scale
Repeatability	(+ or -) 1% of Full Scale on either range
Response	Virtually Immediate in all ranges
Power Supply	6 Volt battery, 4.0 amp hours 120/240 VAC, 50/60 Battery Charger
Controls	Combination Range Switch for: ON/OFF and Range Selection, Reference Adjust
Recorder Output	0-1mA (user adjustable), 100Ω maximum resistance
Reference Standard	0.02 NTU (Nominal)
Dimensions	11" x 9 1/4" x 5 1/2" (27 cm) x (22.7 cm) x (13.5 cm)
Weight	4.5 lbs.(2.05 kilograms)
Operating Temperature	0 - 50°C (32 - 122°F)

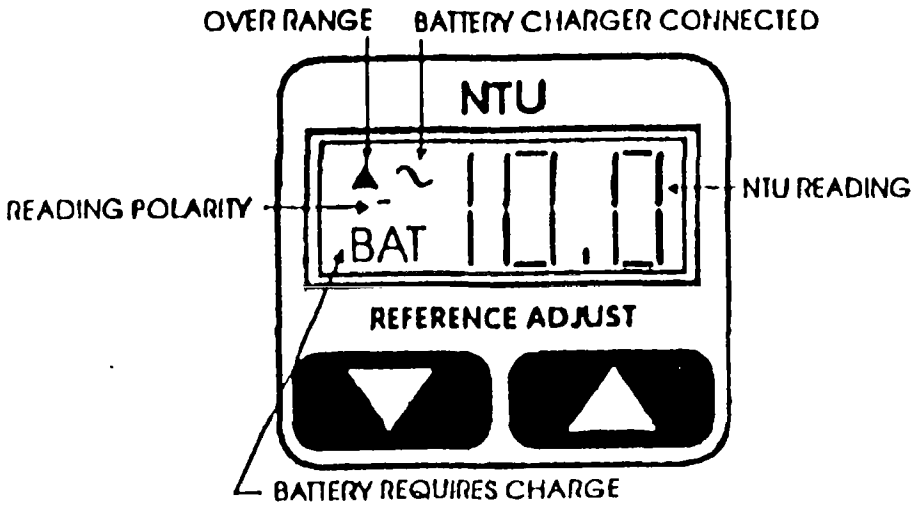
II. PACKING LIST FOR THE DRT-15CE

QTY.	DESCRIPTION
1	Instruction Manual
1	Reference Standard 0.02 NTU (Nominal)
1	Battery Charger 120 V 60 Hz (US style) OR Battery Charger 240 V 60 Hz (European style)
1	Cuvettes complete with screw top (Reorder Cat. #50051)
1	Recorder Plug

A complete listing of spare parts is on page 11 of this manual.

III. PRE-OPERATION CHECK-OUT

Extreme care should be taken when handling the Reference Standard or sample cuvettes as surface scratches or finger smudges will cause analysis errors. Handle these items by the top only.



The battery, when new, usually requires several cycles of discharging and recharging in order to obtain optimum rated life between charges.

The turbidimeter provides up to 20 hours of continuous operation as a portable battery operated unit between recharges.

It is recommended that the unit be turned off between readings in order to obtain longer battery life between recharges. If used as a stationary unit leave the charger plugged in, but TURN OFF INSTRUMENT WHEN NOT IN USE. This will keep the battery at an optimum level at all times.

If for some reason the battery has been completely discharged, the display may not come "on" at all. If this happens turn the unit "off" and recharge the battery with the battery charger. To establish that the unit may be used while charging it, turn the unit on periodically and observe the lower left corner of the display for the word "BAT". When it goes off, (can no longer be seen while the rest of the display is on), the instrument may be used while the charger "tops off" the battery. Depending on the state of discharge of the battery, it may take as long as 6 - 8 hrs. to fully charge it. If the "BAT" indicator is "on", when the charger is not connected, the battery requires charging.

IV. OPERATION AND DESCRIPTION

To operate the turbidimeter, it is first necessary to standardize the instrument. Switch to the "10" range and place the Reference Standard (0.02 NTU) in the optical well.

The EPA recommends that cuvettes used for instrument calibration or sample measurement be indexed. For quick and repeatable indexing of the Reference Standard, an indexing ring and locator pin are included with this instrument.

When shipped, the white locator pin is installed in the collar ring around the optical well of your turbidimeter. The indexing ring is included in the accessory section of this instrument.

To index your Reference Standard, slowly rotate the Reference Standard, at least one complete revolution, while observing the reading, and locate the position of the lowest reading. Without moving the Reference Standard, install the indexing ring over the ridged cap of the Reference Standard, install the indexing ring over the ridged cap of the Reference Standard such that the notch on the ring aligns with the locator pin.

When indexing the Reference Standard in the future, simply insert the Reference Standard and rotate it until the notch on the indexing ring faces the locator pin. Please note that this Reference Standard is only indexed to the turbidimeter for which it was aligned.

To standardize, first index the Reference Standard as above. Then adjust the Reference Adjust in the appropriate direction to cause the display to read 0.02 NTU. The unit is now ready for use on any range.

To make a measurement of a sample, clean one of the cuvettes and fill it to within approximately 1/2" (12 mm) of the top with the sample to be measured. Place the cap on the cuvette and carefully clean the outside surface of the cuvette with a lint free wiper such as "Kimwipes". Place the sample in the well and take NTU reading directly from display. Select the appropriate range for best readability.

If the instrument has been subjected to cold (below 10 degrees Celsius) and then brought indoors, it should be allowed to warm up before use, since condensation may form on the various lenses. Warm up can be aided by leaving the case open and the instrument on for approximately a half hour.

V. RECORDER OUTPUT

The DRT-15CE has a 0-1 mA Recorder Output. The jack is located on right side of the chassis (refer to J2 on figure 2). To use, connect the 1/8" miniplug provided to your recorder. Adjust R17 (pot nearest jack) to obtain a full scale output compatible to a full scale reading on the DRT-15CE. Once this adjustment is made, the DRT 15CE will always be set up for this recorder. Use 10, 100 or 1000 NTU standard in appropriate range.

VI. CRITICAL MEASURING AREA

The critical measuring area of the sample cuvettes is the 3/4" wide band starting 5/8" above the bottom. Keep this area clean and free of scratches or abrasion. Handle by the top part only. (See Figure 1).

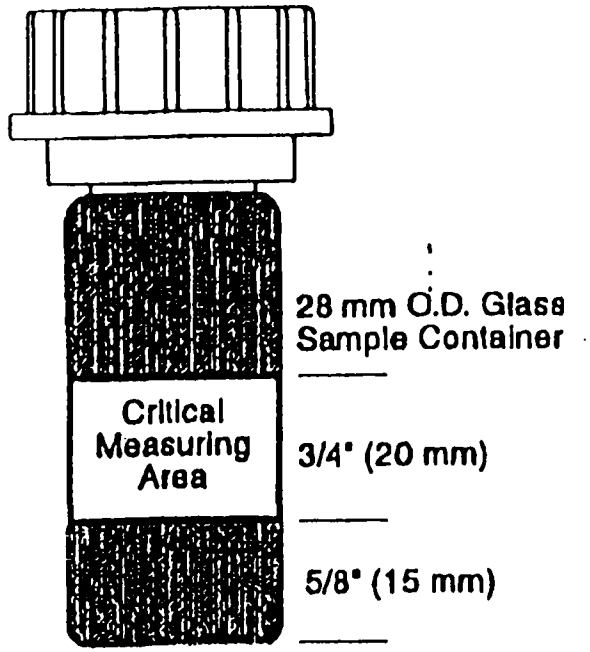


Figure 1

VII. CALIBRATION PROCEDURES

1. Calibration Standards

A. Secondary Standard Set (optional) Catalog No. 19071

HF Secondary Standards are recommended and certified by HF scientific. They are traceable to freshly prepared formazin primary standards. These standards are very easy to use off the shelf anytime without preparation making them an ideal turbidity standard. A Certificate of Traceability is available on request to HF scientific Customer Service Department. HF Secondary Standards may be used for calibration of HF turbidimeters. Order from HF scientific, Inc.

NOTE: Do not freeze standards.
Do not leave standards in the measuring well for extended periods.
Do not shake standards.

Specific instructions for using certified Secondary Standards are included with the kit.

Each Secondary Standard Kit contains:

- Instructions
- 0.02 Reference Standard
- Certified Secondary Standards 10.00, 100.0, 1000 NTU Standards are contained in preselected cuvettes with light shield caps.
- A sturdy storage case

B. Standard Formazin Solutions

Calibration of this instrument is based on Formazin, a material which is made by polymerization.

Calibration samples may be obtained by diluting Formazin stock suspension using "Turbidity-Free" water. Formazin stock suspension can be prepared by the user (Reference Standard Methods For Examination of Water and Wastewater) or a kit can be purchased from, HF scientific, Inc., Catalog No. 50040.

Each kit contains:

-- Instruction manual
-- 1 liter of 4000 NTU Stock Suspension
-- 1 Gallon (3.79 liters) turbidity-free water
-- 4 Sample cuvettes (28 mm)
-- 4 Light Shield Caps
-- Graduated Pipettes 1 ea. in 1 ml, 10 ml, & 25 ml
-- 1 Reference Standard

The following table gives the recommended dilutions of the stock suspension. Be sure to adequately mix the stock suspension prior to removing a portion for dilution.

PROCEDURE

NTU Value	Pipette	Amount	Base Formazin	Volumetric Flask
10.0 NTU	10.0ml	2.5ml	of 4000 NTU	1000ml
100.0 NTU	10.0ml	5.0ml	of 4000 NTU	200ml
1000.0 NTU	25.0ml	25.0ml	of 4000 NTU	100ml

NOTE: 1. When the prepared samples start to flocculate, they are unreliable and fresh ones must be made. This will occur more rapidly for the lower value diluted suspensions. Prepare 40 NTU and lower standards daily.

2. ELECTRONIC CALIBRATION USING FACTORY CERTIFIED SECONDARY STANDARDS (CAT. NO. 19071)

The DRT Turbidimeters have been carefully calibrated at the factory. However, should the Electronic P.C. Board, the Photo Detectors, or the Light Source be replaced recalibration may be easily accomplished at your facility.

To carry out a complete calibration the following Secondary Standard values are required:

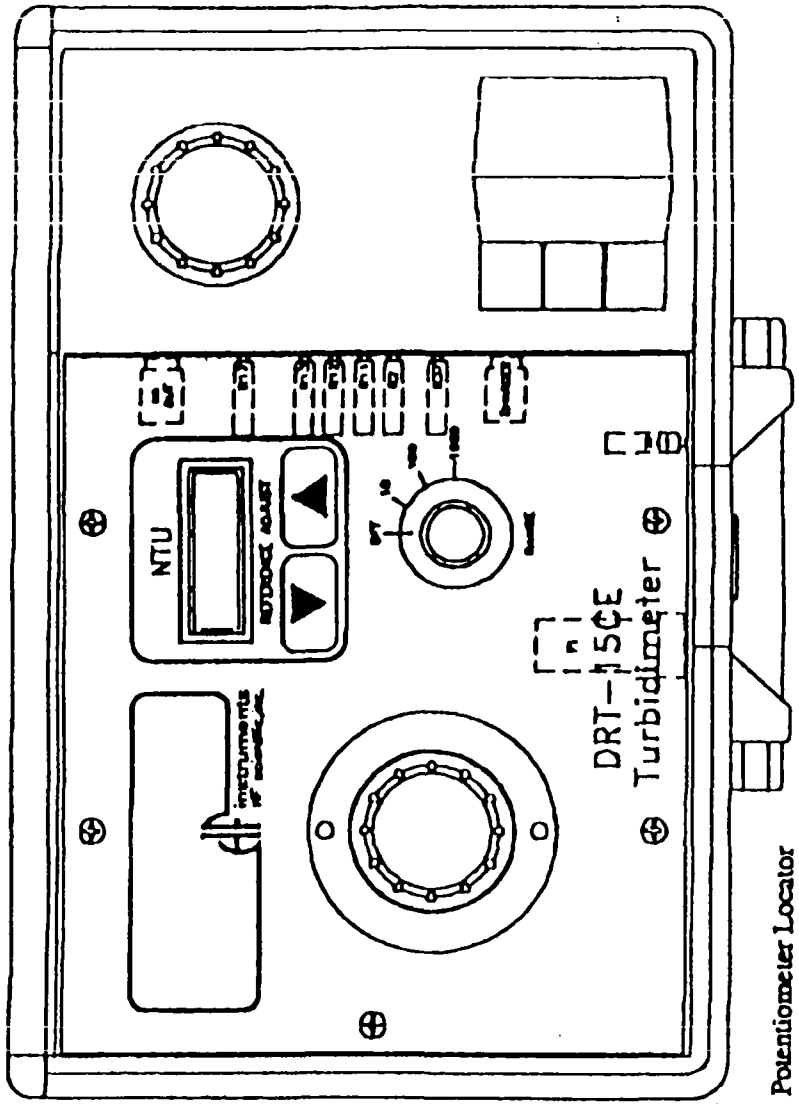
1000 NTU,
100 NTU
& 10 NTU

- Keep the outside surface of cuvettes clean.
- When placing any standards in the well, always use the screw cap/ shield to cover the well in order to keep out ambient light.

To gain access to the trimpots, remove the accessories from the foam holder. Refer to figure 2 for trimpot identification during the next few steps.

1. Turn the DRT-15CE to the 0 - 10 Range.
2. Insert the reference standard and index as described in "Operation and Description".
3. Set the Reference Adjust to the maximum "up" position.
4. Adjust R2, Ref. Offset trimpot, until the display reads 0.18 NTU.
5. Set the Reference Adjust to 0.02 NTU.
6. Insert the 10 NTU standard and index. Adjust R11 (0 - 10 NTU gain) to obtain a reading of 10.00 NTU.
7. Rotate the range switch to the 100 NTU range. Replace the 10 NTU standard with the 100 NTU standard and index.
8. Adjust R12 (0 - 100 gain) to obtain a reading of 100.0 NTU.
9. Rotate the range switch to the 1000 NTU range. Replace the 100 NTU standard with the 1000 NTU standard and index.
10. Adjust R13 (0 - 1000 gain) to obtain a reading of 1000 NTU.
11. It is not necessary to adjust R37 (low battery indicator). This is factory set to indicate a low battery condition when the battery voltage is below 5.6 V.
12. WARNING: R41 is factory set. Adjustment of this trimpot could cause damage to battery and/or DRT-15CE.

This completes the calibration of the DRT-15CE.



Potentiometer Locator
Figure 2

VIII. TROUBLESHOOTING

Symptom

Meter does not respond when a sample is set into the well.

Possible Cause

1. Lamp is burned out. Replace the lamp and recalibrate.
2. Printed Circuit Board failure. Replace Printed Circuit Board.
3. Battery is low. Charge battery.

In the case of 1 or 2 the instrument should be recalibrated. The lamp is an exceedingly long life lamp and therefore replacement is infrequent.

Symptom

Reference Adjust does not have enough travel to adjust for the reference standard value.

Possible Cause

1. Scratched or rubbed reference standard container or aged reference standard. Replace the standard.
2. Optics have aged. Recalibrate.
3. Faulty lamp. Replace the lamp and recalibrate.

Symptom

The display will not stabilize with the reference standard in the well.

Possible Cause

1. Battery voltage too low. "BAT" indicator is "ON" when the battery requires recharging. When the battery is discharged, the voltage will drop off causing the meter display to drift. Recharge battery.
2. Cold sample can cause condensation on the cuvette. Clean and dry cuvette.
3. Unit has not been given sufficient time to stabilize at ambient temperature. Allow time for instrument to stabilize.

Symptom

No display on instrument at all.

Possible Cause

1. Battery is dead. Recharge battery.
2. Fuse has blown. Check battery connections and replace fuse with 1A fast acting 5x 20 mm.

Symptom

Low battery indicator on after 8 hour charge.

Possible Cause

1. Battery is defective. Replace battery.
2. Charger is defective or is not connected to a valid power source. Check for "-" indication on display.

IX. MAINTENANCE

The DRT-15CE is not designed for field servicing. It should be returned to your local distributor or to HF scientific for any service requirements. Be sure to obtain return authorization prior to return. This will facilitate prompt servicing of the unit. The exceptions are Battery and Lamp Assembly replacement. This can be done in the field provided the new battery is installed in the same manner as the battery being replaced.

A. BATTERY REPLACEMENT

Should the battery (HF catalog #70008) fail, it can be user replaced. Make certain the instrument is turned off. Remove all the accessories in the foam holder. Next, remove the foam insert by placing a finger in the cuvette hole and pulling the rear of the foam forward, then up. The five phillips head chassis screws should be removed. Remove the chassis from the case by sliding the chassis all the way to the right then pulling up. The two screws under the right side of the chassis need to be removed now. This will allow for the removal of the battery clamp and the old battery. Replace the battery by reversing the procedures described above. When connecting the battery, be certain that you connect the red wire to the terminal marked (+) and the black wire to the terminal marked (-). If these wires are inadvertently reversed the fuse will blow and must be replaced (see Figure 2).

B. LAMP REPLACEMENT

The lamp source (HF catalog #21084) used in the DRT-15CE has an extremely long life. Before replacing the lamp, make certain that the battery is not in need of a charge and is not defective. Prior to replacing the lamp, remove the battery as described in battery replacement. Remove the lamp wires at terminal block. To remove the lamp, loosen the two set screws on the outermost barrel with a 5/64" hex wrench and pull the lamp straight out. Replace the lamp in the reverse order. Make sure the lamp is pushed all the way in. The set screws should be snugged up; excess pressure could damage the lamp. Replace the battery. Replace the chassis in the case and recalibrate as described in CALIBRATION PROCEDURES SECTION VII, page 5 in this manual.

X. PARTS & ACCESSORIES

HF scientific, inc.
PARTS AND ACCESSORIES FOR
DRT-15CE

CAT. NO.	DESCRIPTION
60002	Reference Standard 0.02 NTU (replace annually)
21805	Instruction Manual DRT-15CE
50051	Cuvettes - Screw Top, 3/pk.
21084	Lamp Source Assembly 2/pk.
70008	Battery - 6 volt
70050	Battery Charger - 120 volt (US style)
20850	Photo Diode
19046	Battery Charger, 240 Volt (European style)
21904	LCD Display
20495	Printed Circuit Board Complete
50040	Formazin Stock Solution Kit
70900	Cuvette Cleaning/Conditioning Solution
20956	Fuse, 1A, 5 x 20 mm
19071	Secondary Calibration Standards - 1 ea.10, 100, 1000 NTU, (Replace annually)
19045	Auto Adapter 12V
50091	Anti-fog kit

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Fort Myers, FL 33916-7597
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XI. WARRANTY

HF scientific, inc., as vendor, warrants to the original purchaser of the instruments to be free of defects in material and workmanship, in normal use and service, for a period of one year from date of delivery to the original purchaser. HF scientific, inc.'s, obligation under this warranty is limited to replacing, at its factory, the instrument or any part thereof. Parts which by their nature are normally required to be replaced periodically, consistent with normal maintenance, specifically lamps, and fuses are excluded. Also excluded are accessories and supply type items.

Original purchaser is responsible for return of the instruments, or parts thereof, to HF scientific, inc.'s factory. This includes all freight charges incurred in shipping to and from HF scientific, inc.'s factory.

HF scientific, inc. is not responsible for damage to the instrument, or parts thereof, resulting from misuse, negligence or accident, or defects resulting from repairs, alterations or installation made by any person or company not authorized by HF scientific, inc.

HF scientific, inc. assumes no liability for consequential damage of any kind, and the original purchaser, by placement of any order for the instrument, or parts thereof, shall be deemed liable for any and all damages incurred by the use or misuse of the instruments, or parts thereof, by the purchaser, its employees, or others, following receipt thereof.

Carefully inspect this product for shipping damage, if damaged, immediately notify the shipping company and arrange an on-site inspection. HF scientific, inc. cannot be responsible for damage in shipment and cannot assist with claims without an on-site inspection of the damage.

This warranty is given expressly and in lieu of all other warranties, expressed or implied. Purchaser agrees that there is no warranty on merchantability and that there are no other warranties, expressed or implied. No agent is authorized to assume for HF scientific, inc. any liability except as above set forth.

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CHLORINE, TOTAL AND FREE (RESIDUAL)

1.0 SCOPE AND APPLICATION

This method is intended for treatment system effluent and is applicable to water supplies, such as chlorinated wastewater effluent, drinking water, groundwater, and polluted water. This method is not applicable to estuarine or marine waters because bromide interferes with the reaction.

2.0 SUMMARY

The chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing microorganisms. In the particular case of chlorine use in the Waukegan Harbor treatment system, it is used to eliminate fouling from bacterial growth in the treatment system. A secondary benefit is the overall improvement in water quality resulting from the reaction of chlorine with ammonia, iron, manganese, sulfide, and some organic substances..

3.0 SPECIAL CONSIDERATIONS

- Analyze immediately. Chlorine in an aqueous solution is not stable, and the chlorine content of aqueous samples or solutions will decrease rapidly. Exposure to sunlight or agitation will accelerate the reduction of chlorine.
- Sample color and turbidity may interfere in all colorimetric procedures.
- Organic contaminants may produce a false free chlorine reading in most colorimetric methods.
- Strong oxidizing agents, such as bromine, chlorine dioxide, iodine, permanganate, hydrogen peroxide, and ozone, interfere in the measurement of free chlorine.

4.0 APPARATUS AND MATERIALS

4.1 Portable Colorimeter, Hanna Instruments, Model HI 93711 (550 nm wavelength LED).

4.2 Sample cuvets, clear colorless glass, scrupulously clean, inside and out. Use separate cuvets for free and total chlorine to avoid iodide contamination in free chlorine samples.

4.3 Free Chlorine and Total Chlorine powder packets.

4.4 Chlorine-demand-free water, such as commercially bottled deionized water.

4.5 Commercially available chlorine and/or potassium permanganate solution standards.

5.0 PROCEDURE

5.1 The Hanna Instruments colorimeter is factory calibrated Turn the meter on with the ON/OFF key, select free or total chlorine by pressing the FREE/TOTAL key, and check the meter following the steps included herein.

5.2 Check the accuracy of the meter by analyzing three freshly prepared potassium permanganate ($KMnO_4$) standards from 0.1 *N* $KMnO_4$ in the range that will be used to measure the samples (0.1 *N* $KMnO_4$= 17,737 mg/L chlorine equivalent). Prepare 0.18, 0.88, and 1.77 mg/L chlorine equivalent standards by diluting 1.00 mL 0.1 *N* $KMnO_4$ into a 1000 mL volumetric flask, then diluting 1.00, 5.00, and 10.00 mL of this solution into separate 100 mL volumetric flasks. Document the calibration check standard readings in a field log.

5.3 Measure each check standard, blank, and sample by filling a clean cuvet to within approximately three-quarters inch of the rim with 10 mL of unreacted sample. Place the cap on the cuvet, carefully clean the outside surface with a lint-free wipe, place the cuvet in the cell and assure that the notch is positioned securely into the groove.

5.4 Press the ZERO key and wait a few seconds for the display to read 0.0 to compensate for any color or turbidity in the sample.

5.5 Remove the cuvet and add the specific test reagent (one packet of HI 93701 DPD Free Chlorine reagent for free chlorine or one packet of HI 93711 DPD Total Chlorine reagent for total chlorine). Replace the cap and shake gently.

5.6 Reinsert the cuvet into the meter. Press the READ key to measure free chlorine, or wait 2 min. and 30 seconds, then press the READ key to measure total chlorine.

5.7 Record the Free Chlorine or Total Chlorine to the nearest 0.01 mg/L.

6.0 FIELD LOG DOCUMENTATION

- Instrument used (currently utilizing a Hanna Instruments colorimeter, Model # HI 93711)
- Date
- Check standard reading(s)
- Chlorine range(s) used (0.00-2.50 mg/L free; 0.00-3.50 mg/L total chlorine)
- Sample (free and total) chlorine readings
- Identification of person performing free and/or total chlorine measurements

7.0 PRECISION AND ACCURACY

Precision and accuracy for this method have not been determined.

8.0 REFERENCES

APHA, AWWA, WPCF, Standard Methods for the Examination of Water and Wastewater, 18th Edition, pp. 4-36 to 4-48, Method 214 (1992).

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Method 330.5 (1979).

Hach Water Analysis Handbook, 2nd Edition pp. 161-198, Hach Methods 8021 and 8167 (1992).